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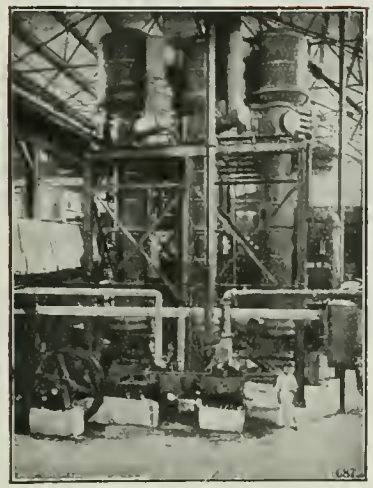

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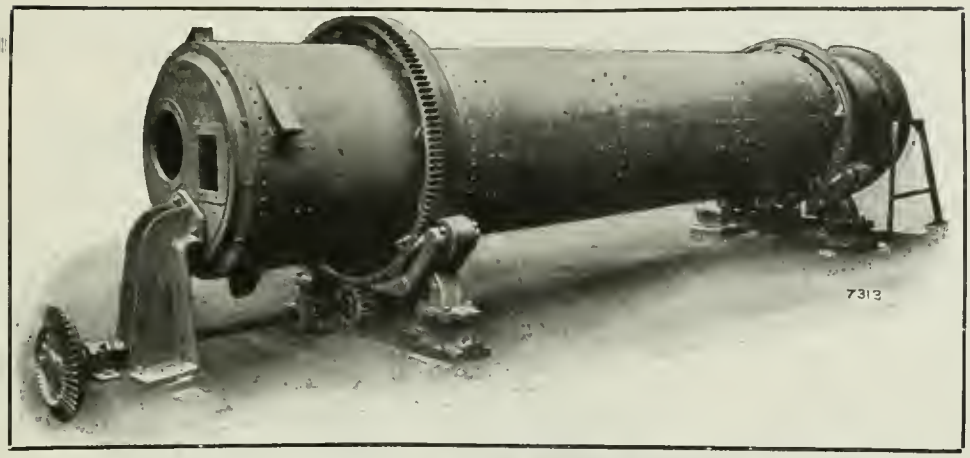

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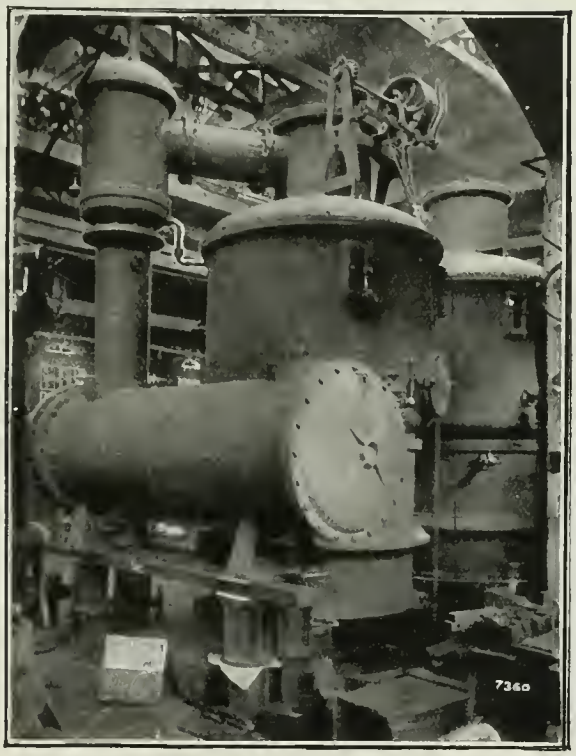

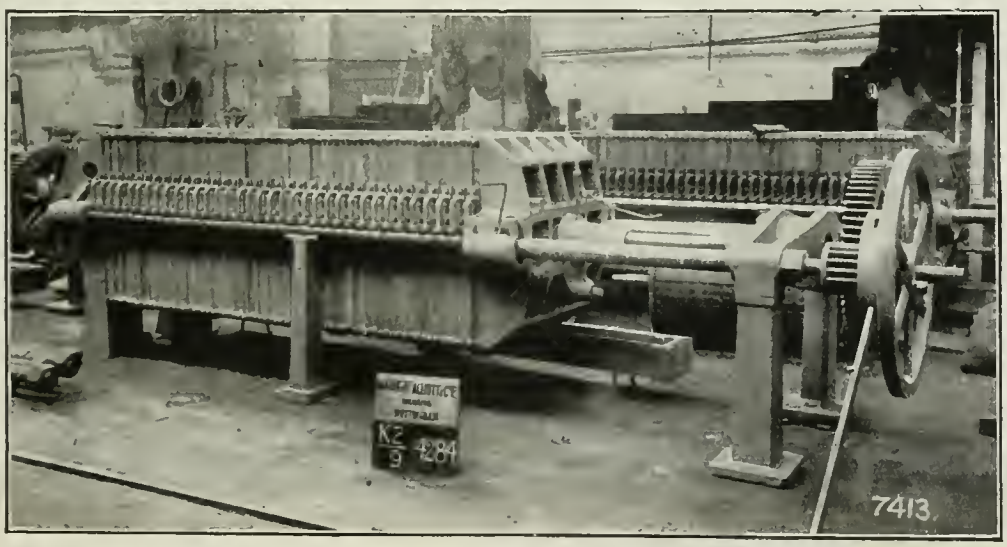

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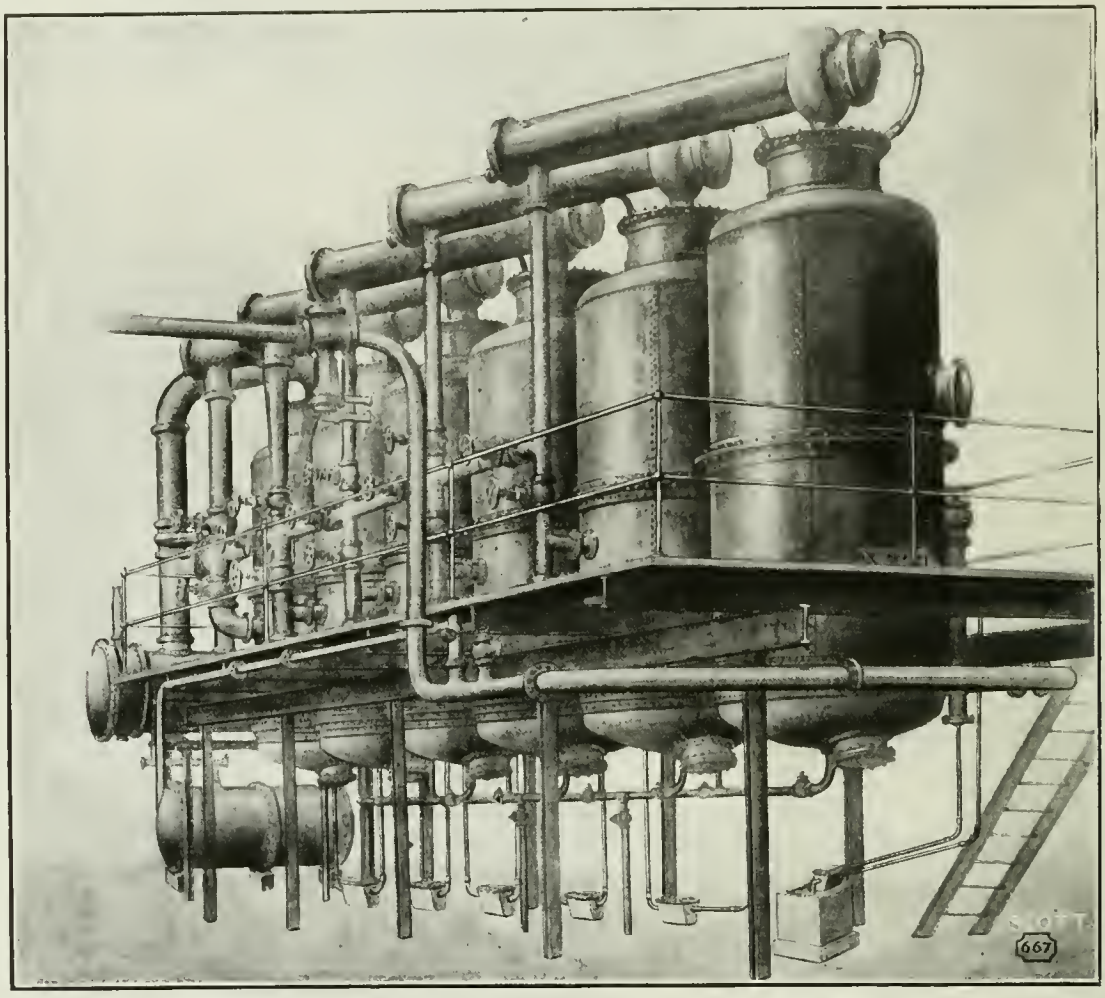

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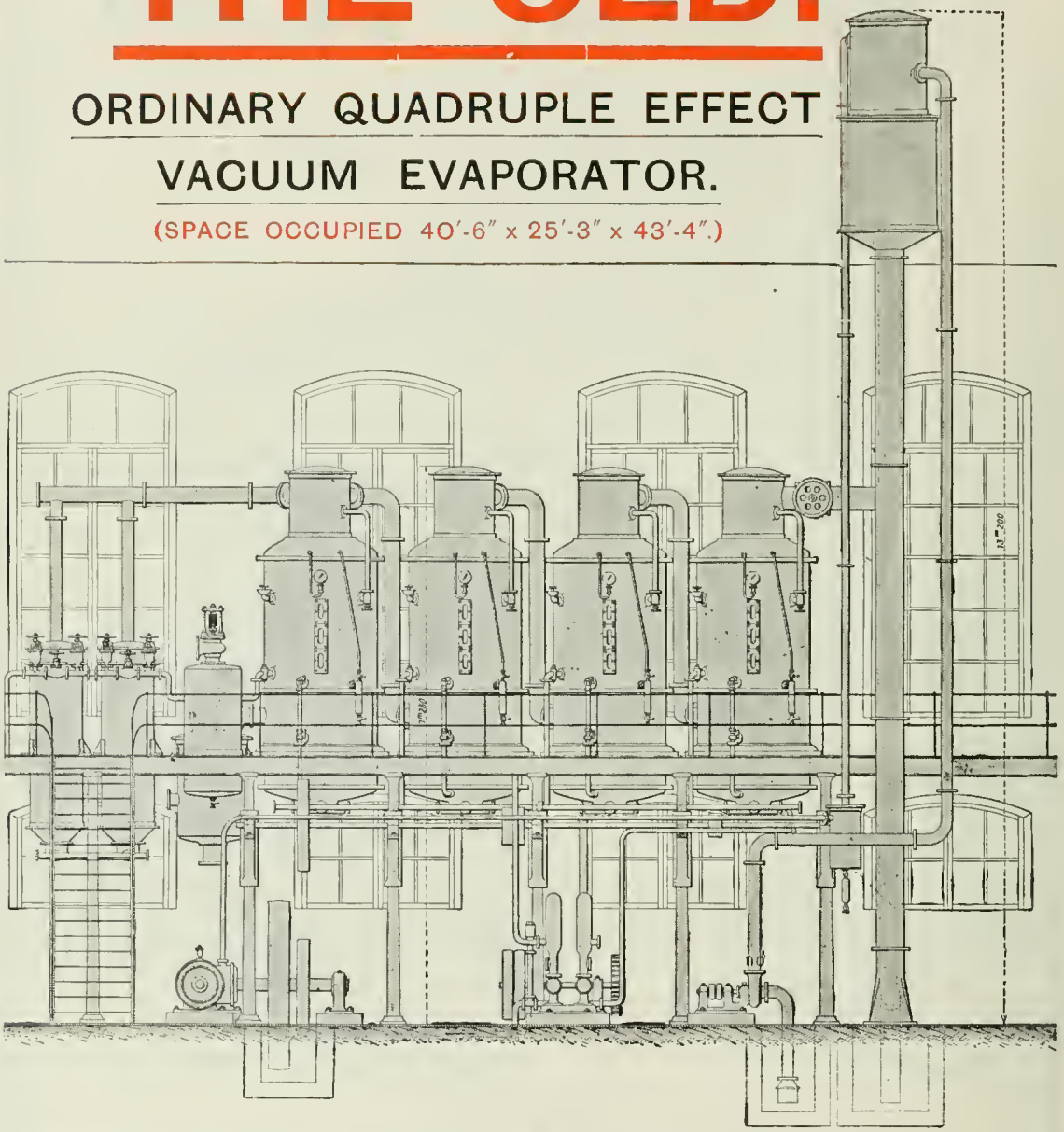

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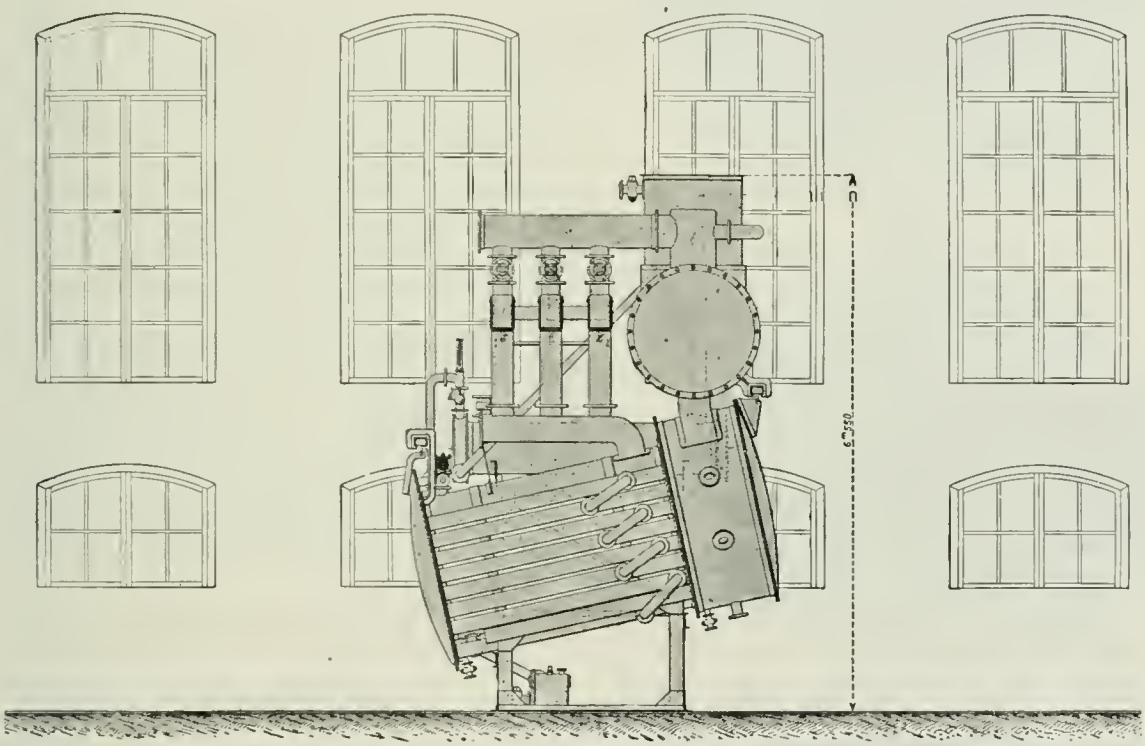

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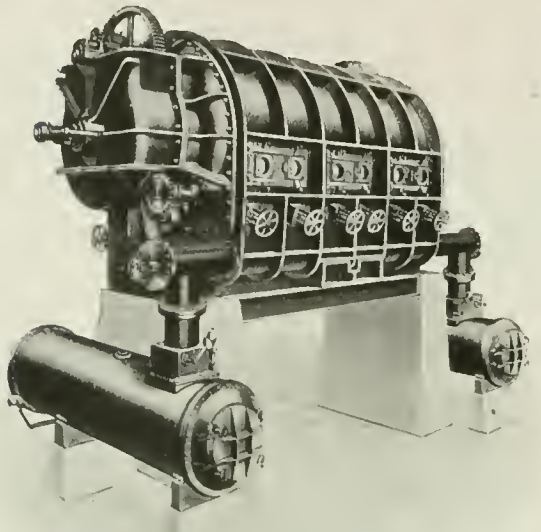

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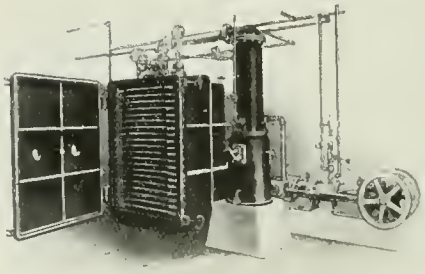

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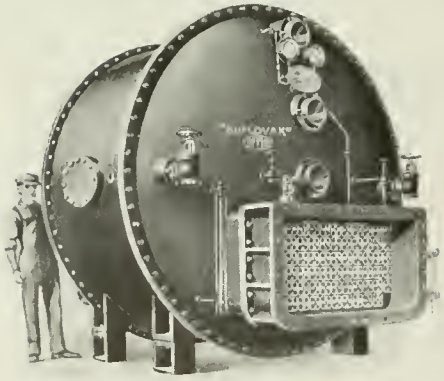

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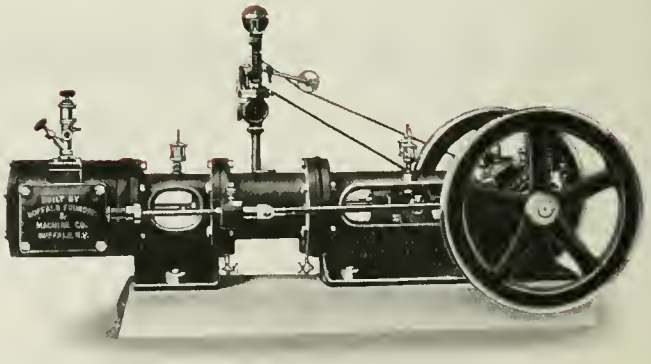

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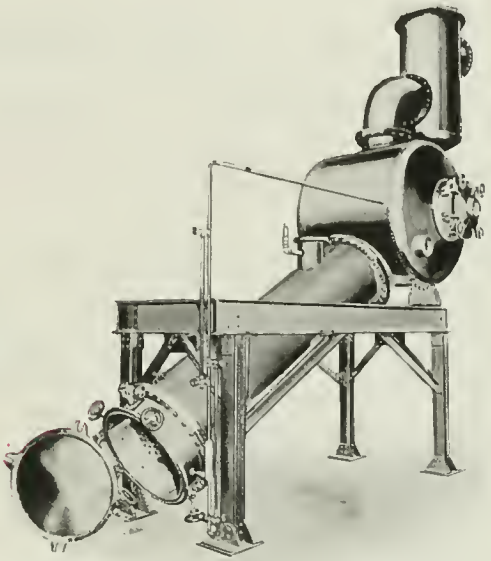

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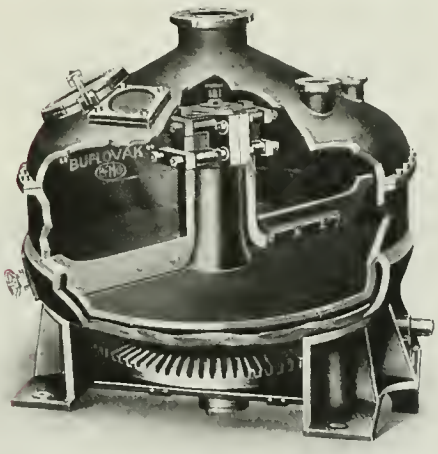

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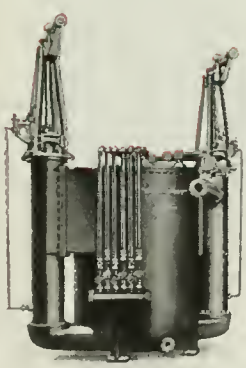

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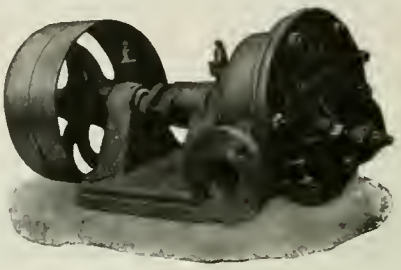

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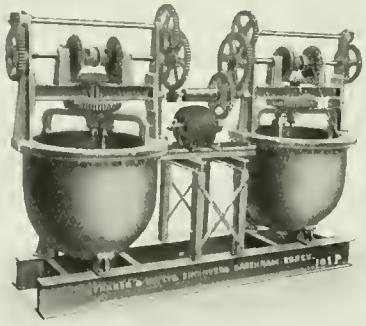

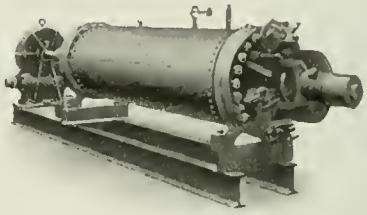

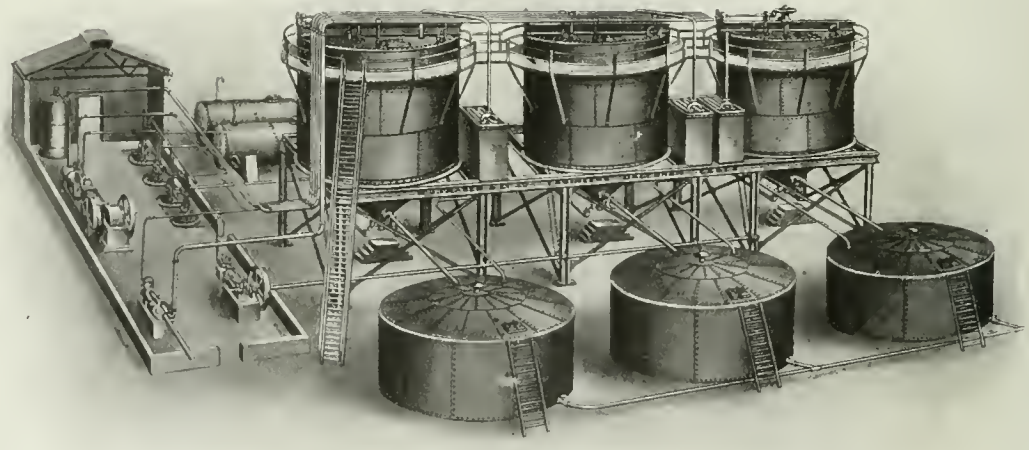

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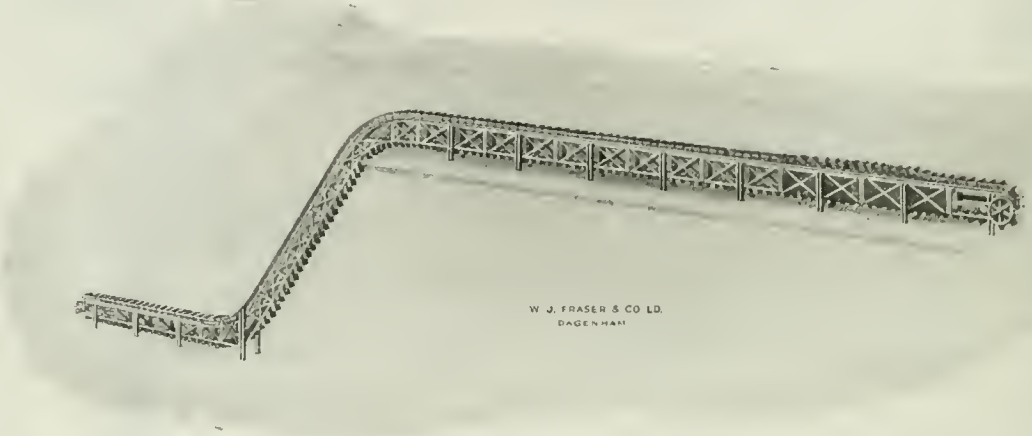

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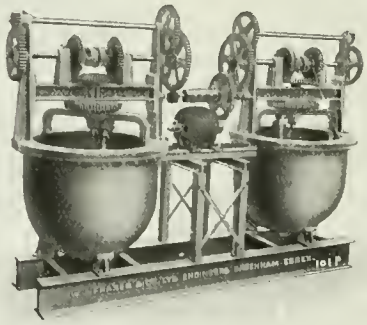

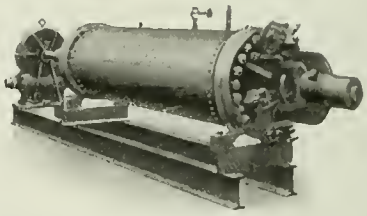

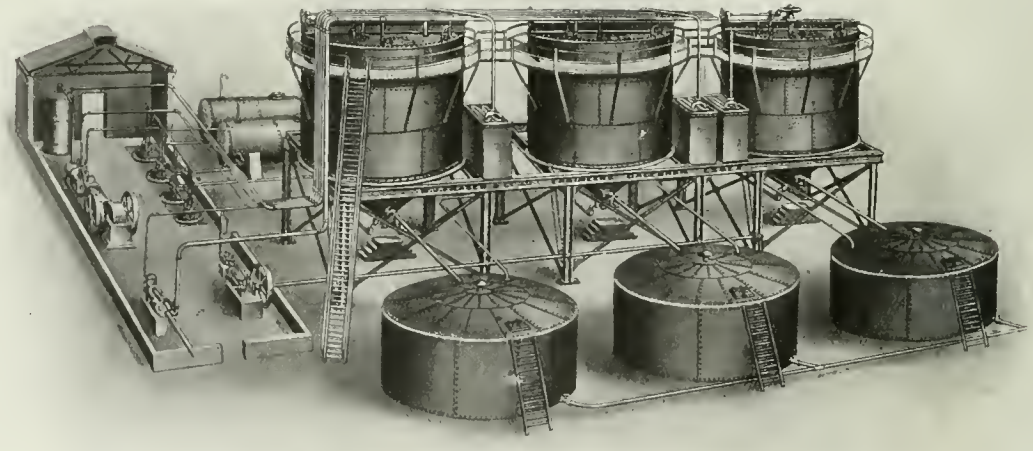

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J. ARTHUR REAVELL, M.I.M.E., M.I.Chem.E.

SOCIETY OF CHEMICAL INDUSTRY

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VOLS. III & IV

1921—1922

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Contents

1921.

PAGE

Plant for the Treatment of Industrial Waste Waters – – *By E. V. Chambers,* 7

The Industrial Applications of Electrical Precipitation Methods *By Dr. H. J. Bush* 25

The Claude Synthetic Ammonia Process and Plant – – *By J. H. West, B.Sc.* 33

De-gassing and Purification of Boiler Feed Water – – – – – – 41
By M. Paul Kestner, President of the Société de Chimie Industrielle.

1922.

Potential Developments in Refrigeration *By Dr. W. R. Ormandy and E. C. Craven* 68

FIFTH CONFERENCE.

EVAPORATION AND DISTILLATION.

Observations on the Design of Ammoniacal Liquor Stills – *By P. Parrish, A.I.C.* 80

Tar Distillation – – – – – – – – – – *By W. A. Walmsley* 110

The General Problem of Evaporation – – – – – – – – – 126
By Prof. J. W. Hinchley, Wh.Sc., A.R.S.M., F.I.C., M.I.Chem.E.

Glycerine Distillation – – – – – – – – – *By T. H. Gray* 136

The De-watering of Peat by Pressure – – – – – – – – – 141
By Prof. J. W. Hinchley, Wh.Sc., A.R.S.M., F.I.C., M.I.Chem.E.

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The Third and Fourth Annual Volumes of Proceedings

OF THE

CHEMICAL ENGINEERING GROUP OF THE SOCIETY OF CHEMICAL INDUSTRY

1921—1922

The Volumes of Proceedings of the Chemical Engineering Group for the years 1921 *and* 1922, *issued annually prior to* 1921, *are published together for the years mentioned, partly for economic reasons and partly because the matter to be included in the volume for each year separately is relatively smaller than usual. The Group Committee propose to adopt this plan in future whenever circumstances seem to render such a course advisable.*

The review given hereunder of the work of the Chemical Engineering Group subsequent to the issue of Volume II of its Proceedings, covers the years 1921 and 1922, and is a record of continued effort, under circumstances of extraordinary difficulty, towards success which was in large measure achieved. The close of the year 1920 was in England synonymous with a phenomenal trade slump, which was continued in greater severity during 1921, and to a large extent during 1922. Consequently, the Chemical Engineering Group, like many similar Scientific Societies, experienced a pronounced diminution in its membership, and bad perforce to curtail many of its normal activities owing to lack of funds. The falling off in subscriptions was not severe during 1921, as payment is due on January 1 in any year, and consequently the revenue from members was only 5% less than the figure for 1920. In 1922, however, the full force of the trade depression became apparent and expressed itself as a 20% drop in the Group's membership and in the income derived therefrom. It became necessary to alter the Group's programme to anticipate the new conditions, and the Conferences of former years were replaced in 1921 by a number of smaller meetings as specified later. This course was adopted for the further reason that the Society's Annual General Meeting, at which in former years the Group had arranged a Conference, was in 1921 held in Canada.

In this year likewise (1921) arrangements were made by the Group Committee with the Council of the Society for a closer co-operation of the new bodies, based upon the previous two years' work of the Group and experience of its needs. Subsequently (in 1922), further steps were taken towards co-ordination of organisation, and the year of issue of this volume (1923) is noteworthy as showing a re-establishment of the membership of the Group at very nearly its original figure, and the consequent placing of its finances upon a satisfactory basis. The Committee of the Group cannot express with sufficient emphasis to the Chairman and Hon. Treasurer of the Group, and to the President and Hon. Treasurer of the Society, their thanks for bringing about this happy result.

The issue of Data Sheets by the Committee in the period reviewed has progressed only slowly, owing to lack of funds. Two new sheets, denominated respectively—

"THE PROPERTIES OF TAN EXTRACT LIQUORS"

and

"CONDENSATION CURVES FOR HYDROCHLORIC ACID"

have been issued as a result of a special grant made by the Council of the Society of Chemical Industry from the Messel Memorial Fund at its disposition. Other sheets are in preparation.

The meetings of 1921 are divisible into two categories: —

(*a*) Propagandist,
(*b*) Scientific.

In the first category are to be placed the following:

(1) Meeting in the Institution of Shipbuilders and Engineers, 39, Elmbank Crescent, Glasgow, February 11, 1921. Chairman, Dr. J. H. Young, Chairman of the Glasgow Section of the Society. The meeting was attended and addressed by (*inter alios*) the Chairman and Hon. Secretary of the Group.

(2) Meeting and Dinner at the Boulogne Restaurant, Lisle Street, London, when the subject of

" CO-ORDINATION OF EFFORT IN CHEMICAL ENGINEERING "

was discussed. A number of distinguished chemical engineers took part in the discussion, the Chairman of the Group presiding.

The Scientific Meetings were as follows: —

1.—" PLANT FOR THE TREATMENT OF INDUSTRIAL WASTE WATERS "

By E. V. Chambers

Paper read in the Huddersfield Technical College, February 24, 1921. Chairman, Dr. H. H. Hodgson, Professor of Applied Chemistry, Huddersfield.

2.—" THE INDUSTRIAL APPLICATIONS OF ELECTRICAL PRECIPITATION METHODS "

By Dr. H. J. Bush

Paper read at a joint meeting of the Chemical Engineering Group and the Manchester Section of the Society of Chemical Industry in Manchester, November 11, 1921. Chairman, J. Allan, Esq., Chairman of the Manchester Section.

3.—" THE CLAUDE SYNTHETIC AMMONIA PROCESS AND PLANT "

By J. H. West, Esq., B.Sc.

Paper read at a joint meeting of the Chemical Engineering Group and the Institution of Mechanical Engineers, held in the Institution of Electrical Engineers, London, November 22, 1921. Chairman, J. Arthur Reavell, Esq., M.I.M.E., Chairman of the Chemical Engineering Group.

4.—" DE-GASSING AND PURIFICATION OF BOILER FEED WATER "

By M. Paul Kestner,

President of the Société de Chimie Industrielle de France.

The propagandist meetings are not recorded in this volume, but the scientific papers are reprinted, together with discussions, as far as reported.

The paper by Dr. Bush at Manchester was extremely successful, securing almost a record attendance, and drawing forth quite an interesting discussion. The possibilities of application of electrostatic forces were relatively unknown in England before the Great War, and Dr. Bush's paper drew for illustration upon a great number of examples of plant erected at munitions factories in England and elsewhere. It is safe to say that the paper was one of the most successful ever delivered before the Manchester Section of the Society.

The joint meetings with the Institution of Mechanical Engineers are invariably successful, and the two papers included in the present volume, and read under the dual auspices, were of outstanding interest and importance. The one by M. Kestner bids fair to become a classic, and the thanks of the Group are due to the President and Council of the Institution in question for their unqualified assent to the reprinting of the paper and discussion.

Following a paper on "Potential Developments in Refrigeration," by Dr. W. R. Ormandy and E. C. Craven, Esq., read in Glasgow in January, 1922, the principal meeting of 1922 was the Conference at Glasgow on the occasion of the Annual General Meeting of the Society of Chemical Industry. The subject discussed was the extremely important one of

" EVAPORATION AND DISTILLATION,"

and four papers (as given later) were read. The attendances at the two sessions of the Conference were extremely good, and the Group owes a meed of thanks to the Glasgow Section of the Society and likewise to the Council of that body for financial help enabling the necessary printing and organisation to be carried out. The paper on

" AMMONIA STILLS,"

by Mr. Parrish, is a monumental work, embodying the author's many years' experience in this class of distillation, and gave rise to a most interesting discussion. The paper is very fully illustrated, and will well repay study by anyone interested in the general problem of distillation as well as in its particular application to ammonia manufacture.

The last paper read before the Group in 1922 was given in London by a former Chairman, Professor J. W. Hinchley, at an informal meeting held at the Chemical Industry Club on November 10, 1922. The subject chosen was one in which the author had done much pioneering work, the narration of which was of absorbing interest, and gave rise to much discussion. Informal meetings of this kind in London are being continued as part of the Group's normal activities.

The Committee of the Group are pleased to reflect that owing to the help given to the Group by the Council of the Society of Chemical Industry on the one hand, and by its members on the other, they have been able to continue their work hitherto without a repetition of those appeals for donations which met with so generous a response in 1919 and 1920. They believe that conditions are improving, and that the Group can look forward to increasingly useful activity, influence, and membership in the future.

LIST OF OFFICERS AND COMMITTEE, 1921.

The Committee and Officers of the Chemical Engineering Group and the names of the Honorary Correspondents or Local Sectional Representatives for 1921 are given below:—

Chairman: J. Arthur Reavell, Esq., M.I.M.E., M.I.Chem.E.

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Edinburgh: J. W. Romanes, Esq., B.Sc.

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Liverpool: A. Holt, Esq., M.A., D.Sc., F.I.C.

London: A. E. Berry, Esq.

Manchester: Howard Cheetham, Esq.

Nottingham: H. Calam, Esq.

LIST OF OFFICERS AND COMMITTEE, 1922.

The Committee and Officers of the Chemical Engineering Group and the names of the Honorary Correspondents or Local Sectional Representatives for 1922 are given below:—

Chairman: J. Arthur Reavell, Esq., M.I.M.E., M.I.Chem.E.

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A. J. Brodghall, M.I.Chem.E.

Prof. F. G. Donnan, F.R.S.

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W. J. Gee, M.I.Chem.E.

Prof. J. W. Hinchley, Wh.Sc., A.R.S.M., F.I.C., M.I.Chem.E.

R. R. Howroyd.

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W. R. Ormandy, D.Sc.

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Bristol: A. Marsden.

Edinburgh: J. W. Romanes, B.Sc.

Glasgow: Jas. Macgregor, M.I.Chem.E.

Liverpool: W. E. Gibbs, D.Sc.

London: R. J. Colgate.

Manchester: H. Cheetham.

Nottingham: H. Calam.

PLANT FOR THE TREATMENT OF INDUSTRIAL WASTE WATERS.

By E. V. CHAMBERS.

(*Paper read at a meeting of the Chemical Engineering Group in the Technical College, Huddersfield, February 24th*, 1921. *Dr. H. H. Hodgson, Professor of Applied Chemistry in the College, in the Chair.*)

The chair at the meeting was taken by Dr. H. H. Hodgson, of the Technical College, Huddersfield, in the unfortunate absence, owing to illness, of the Chairman of the Chemical Engineering Group, Mr. J. Arthur Reavell. Dr. Hodgson commented upon the importance of the work being done by scientific societies, such as the Society of Chemical Industry and the Chemical Engineering Group, in spreading useful knowledge applicable to the development of industry, and said the Group in particular was to be commended for its enterprise in holding its meetings in various parts of the country instead of centralising, as was the very common practice with other societies. He then called upon Mr. Chambers to read his paper.

Introduction.

The term "waste water" is somewhat significant. Originally it may have been intended for these liquids resulting from various manufacturing processes, which at the time could be put to no further use. To-day the term has assumed a fuller meaning. By modern processes, many materials formerly classified as waste, have by their recovery become sources of national wealth and individual profit. This exploration of waste has extended into the domain of factory effluents, where rapid progress is being made, and before long, in certain manufacturing areas, even the purified effluent, unless reused, will be regarded as waste in the real sense.

Two factors have contributed to the awakening in regard to the prevention of waste in industrial effluents—the first being the necessity for more efficient production under pressure of competition, and the second, the Rivers Pollution Act. A large measure of recognition is due to the Rivers Board Authorities for the part they have filled in the prevention of national waste. It will be true to say that in certain industrial areas the *administrative* cost of the Rivers Pollution Authorities has been repaid a hundredfold as the direct result of their vigilance, supervision and assistance.

There is a general similarity in all effluents which result from the same trade process. In the woollen industry, for instance, there is little material difference between the effluents resulting from similar processes in the various factories.

The limitation of time makes it impossible to deal effectively with the many processes of effluent purification. The examples of a few different types will, it is hoped, give some indication as to the nature of the work involved.

The woollen industry is not the least important of our national industries. It is represented by centres in the West Riding of Yorkshire, the border counties of Scotland, the West of England, parts of Lancashire and also in Ireland. The various manufacturing operations from the raw wool to the finished cloth are seldom carried through in one and the same factory. The industry is subdivided into specialised sections, namely:—

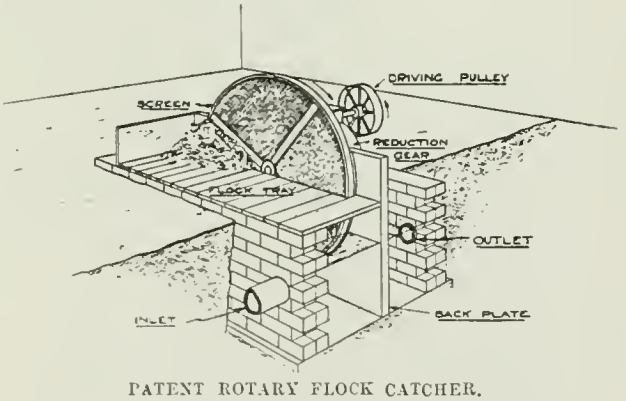
1. Wool scouring, carbonising and combing.
2. Spinning.
3. Weaving.
4. Dyeing and finishing.

These processes apply to the raw wool as it is imported. In addition there is the shoddy industry, where worsted and other rags are torn up into a loose fluff and re-spun and converted into cloth again. Shoddy is also submitted to the scouring, dyeing and finishing process. As the rags from which shoddy is made may contain cotton materials the process of carbonisation is resorted to in order that the cotton may be eliminated.

In the woollen industry several distinct effluents are produced. These are:—

1. Raw wool washing effluent.
2. Yarn and piece scouring effluent.
3. Effluent from the various dyeing processes.

While different methods of treatment must be adopted for each distinct type of effluent, there is one preliminary process common to all, namely, the careful screening of the effluent for the recovery of valuable fibre which is invariably present. Before describing the different methods of purification it may be advisable to deal with the screening at this stage. Formerly this was attempted by allowing the effluent to flow through fixed screens composed of wire mesh or perforated plates. The mesh or perforations varied from $\frac{1}{16}$" to $\frac{1}{4}$" diameter. As the wool fibre has a maximum diameter of one-fiftieth of



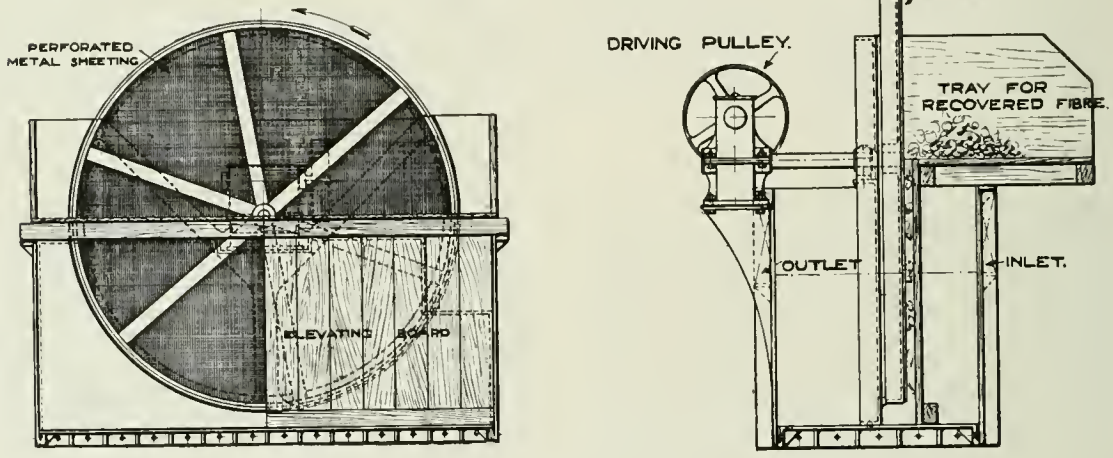
PATENT ROTARY FLOCK CATCHER.

FIG. 1.

an inch it will be readily understood that many of the fibres travelling parallel with the flow would thread their way through the perforations. Even those fibres which were retained by the screen eventually choked the perforations and thus prevented the free flow of the effluent with consequent trouble in the drains and channels.

Figure 1 represents a successful automatic screen which effectively removes all the fibre without retarding the flow of effluent. The screen consists of a perforated disc which rotates on a horizontal axis. The disc fits into a machined backplate so constructed that the effluent must pass through the perforations which form the screen. It is mounted on a shaft between two bearings. The screen revolves slowly and is therefore fitted with suitable reduction gearing.

Figure 2 is a sectional drawing of the screen. The screen is fixed either in a cast iron tank or in a brickwork chamber, through which the effluent flows. Only about one-third of the diameter of the screen is immersed in the effluent. It has been stated that an individual fibre can thread its way through a fixed screen, but it cannot do so in the rotating disc. In the screen tank the velocity of the effluent is reduced to a flow of one inch per second. The velocity of that portion of the disc which is immersed is never less than six inches per second. Therefore before a fibre has travelled one-sixth of its length through a perforation it will come in contact with the edge of the perforation where it is securely held until subsequently removed on its upward traverse. Other fibres, including small



Front Elevation. End Elevation.

FIG. 2.

bunches of fibre conveyed by the effluent, accumulate in front of the screen and a means is provided for raising these fibres out of the effluent. Close to the face of the screen at the bottom right hand quarter is fixed a plate parallel to the face of the screen, leaving an intervening space of 1″. (Front elevation.) The accumulated fibre finds its way between this plate and the screen, and it is at once carried to the top of the plate by the upward traverse of the disc, and is delivered on to a receiving table. The recovered fibre remains in rubbing contact with the face of the screen and thus withdraws and recovers from the screen the individual fibres which had partially threaded the perforations. It will be understood that no cleaning mechanism is necessary, the recovered fibre being very effective for this purpose. This type of screen is built in various sizes ranging from 3 ft. diameter to 10 ft. diameter, for respective flows of 4000 gallons per hour and 60,000 gallons per hour.

Figure 3 is a larger screen 10 ft. diameter at work in a woollen mill. Figure 3A gives another view of the same machine. A large number of screens of this type is at work upon wool, cotton, paper and other effluents, and it is useful in recovering various kinds of suspended solids from effluents. Owing to the fact that the fibre is lifted from the effluent and squeezed before its arrival on the receiving table, it is found to be in a clean condition and of better quality than that recovered by the fixed screen. Partly by actual weighing and partly by estimation it has been ascertained that in 1920 one and a-half million pounds of useful wool fibre were recovered by this type of screen, most of which was made up into yarn and cloth. Apart from the value of the recovered fibre it should be said that its removal from the effluent reduces the ultimate quantity of the solids to be dealt with, and also prevents the choking of drains, pipes or channels which invariably occurs when the fibre is not previously removed.

Various Methods of Purification.

Distinct methods must be adopted for different types of effluent. Pressure or gravity filtration will suffice for liquids of a non-colloidal character, where it is desired to remove only suspended solids. The same result may be accomplished by the use of centrifugal machines, and in one instance by an ingenious combination of filtration and centrifugal force, manufactured by Centrifugal Separators, Ltd., London, and introduced by W. J. Gee, the maximum filtering efficiency is maintained continuously, while the solids are recovered in a convenient and compact form.

When colloidal substances are to be removed from effluents, or dissolved organic or inorganic bodies, it is often necessary to adopt some form of chemical treatment. The more commonly used reagents are lime, ferrous sulphate, (copperas) crude aluminium sulphate, (aluminoferric) and sulphuric acid.

The biological process has not been extensively applied to the purification of industrial waste waters. Certain effluents which, even after a substantial chemical treatment contain putrefactive nitrogenous bodies, can be effectively dealt with by percolating biological filters, so that the stream into which such effluent is discharged is not robbed of its free oxygen content.

Waste waters from coal by-product recovery plant, and particularly those from the ammonia stills are very difficult to purify. When waste steam is available in sufficient quantity evaporation appears to be the only effective solution.

Some details will now be given of a purification

and recovery plant for the treatment of an effluent from a woollen cloth manufacturing establishment. Figure 4 is a photograph of an installation for the purification of effluent from a woollen cloth or piece scouring establishment. Woollen cloth as it comes from the loom contains a considerable percentage of oil. This oil may have been added to the rags or to the loose wool during the tearing up or the blending process previous to spinning. The oil acts as a lubricant, facilitating the operations of carding, blending, spinning and weaving. The percentage of oil may vary considerably according to the quality of material being manufactured. Low grade materials contain a larger proportion of oil than

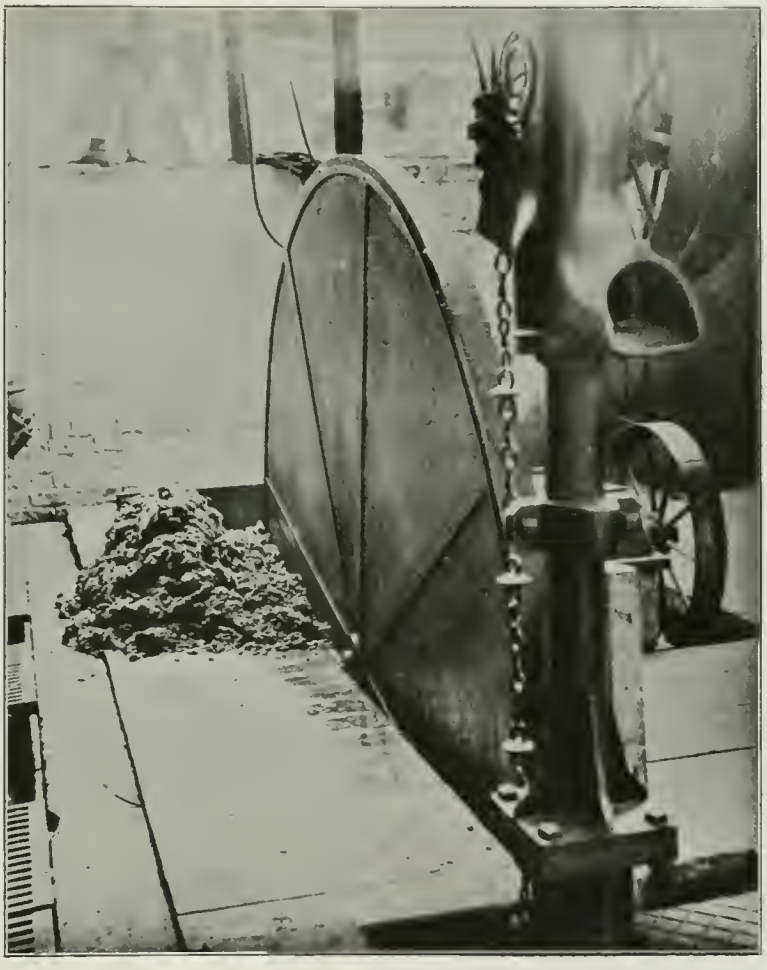


Fig. 3.

the better quality fabrics. It is frequently found that a medium quality cloth contains 9 by weight of oil. The object of the scouring process is to remove this oil and cleanse the cloth. The quality of the oil ranges from that of a high grade oleine containing 98% free fatty acids, down to an oil containing 60% free fatty acids.

The materials used for scouring are soaps, soda ash and various more or less useful scouring assistants. The cloth pieces, in an endless band, pass between heavy wooden rollers and then into the bottom of the scouring machine where they become saturated with the soap and soda solution. The alternative squeezing and soaking continues until the whole of the free oil has been removed from the cloth, by which time the soap solution has been converted into a thick emulsion. This emulsion is then discharged from the machine and simultaneously water is admitted to the machine in the form of a spray which is directed upon the cloth as it continues its cycle in the scouring machine. This washing continues until the effluent from the bowl is free from emulsified grease. The scouring process is then complete.

The effluent from this process contains the whole of the soap and soda used for scouring, together with the oil originally contained in the cloth. In the purification process it is the practice to separate this liquor into two portions. The first portion discharged from the scouring machine contains the greater quantity of the soda ash and soap and oil, and is therefore directed to a separate receiving tank or vessel. The second portion contains smaller quantities of scouring liquor and wash water and its treatment will be referred to subsequently.

On the average it may be stated that a "piece" of cloth is 60 yards long and weighs 60 pounds. Each "piece" of cloth requires 500 gallons of water for scouring, only one-third of which (the first portion of the waste scouring liquor) or 166 gallons is

treated for oil recovery. The following figures represent those of a factory scouring 60 "pieces" per day.

Total weight of cloth	3,600 lb.
Oil content 9% plus 65% of soap used for scouring (3 lb. per piece)	441 lb.
Soda ash (4 lb. per piece) (used for cleansing) ..	240 lb.
Water to be treated for recovery of oil (60 pieces at 166 galls.)	10,000 galls.

The treatment process consists of the addition of sulphuric acid to the scouring liquor which neutralises the soda carbonate and sets free the oil. After a period of settlement the more or less clear acid water is decanted and the grease or magma is forced through steam heated filter presses where the oil is separated from the magma.

In Figure 4 the plant is designed to deal with 22,000 gallons of strong scouring liquor per day. The method of working the plant is as follows.

wagon. It is pumped into an overhead tank by means of an all iron hand pump. From the overhead tank it flows into measuring vessels constructed of cast iron, and from thence a measured quantity is allowed to enter the tank. To each tank of scouring liquor is added sufficient sulphuric acid to decompose the sodium carbonate and to give the liquid a slightly acid reaction. It is found that the treated liquid must have an acidity equal to 20 parts per 100,000 before complete separation takes place. At this point the emulsion is entirely broken down and the grease or oil is liberated and forms a heavy scum or magma in the tank.

Agitation.

A system of mixing is necessary to bring the acid into intimate contact with the liquid. This mixing may be done either by means of mechanical agitators,

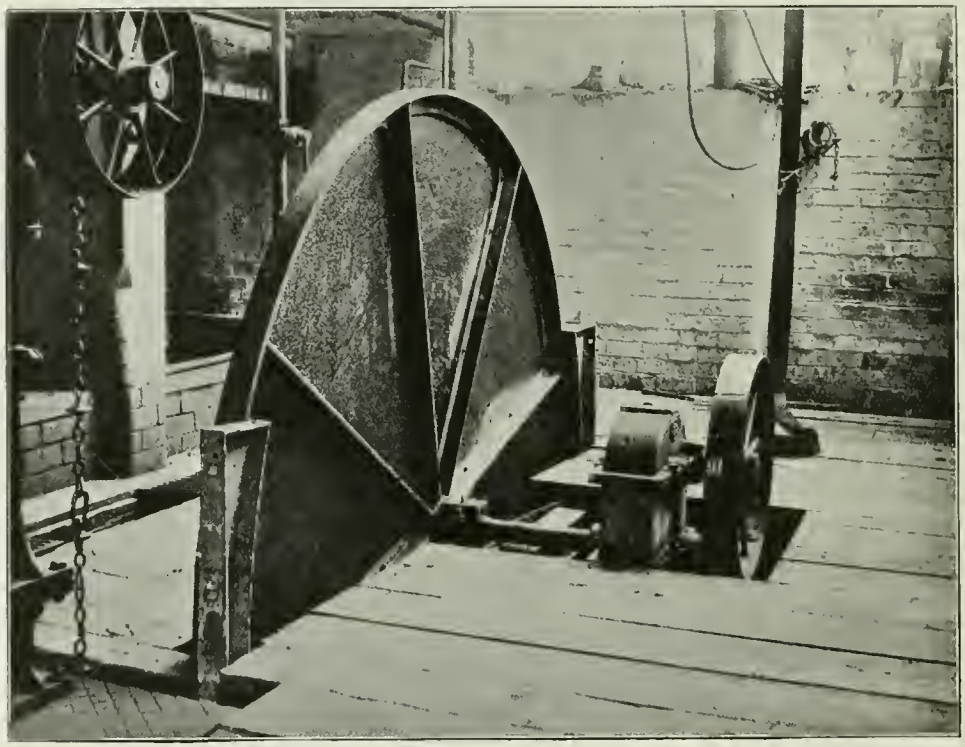


Fig. 3A.

Screening.

A 4 ft. diameter screen first removes the fibre which is collected for re-use. Unless the fibre is removed it causes trouble in the pumping operation. It chokes the valves in reciprocating pumps and attaches itself to the impellers in centrifugal pumps.

Pumping.

The type of pump will depend upon local conditions. It may be steam, belt, or electrically driven, and both ram, piston pattern, and centrifugal pumps are used. The pumping of the screened liquor presents no difficulties. An all iron pump is the most suitable, as the scouring liquor, owing to its content of grease, has a softening and disintegrating action on vulcanite and other "composition" valves.

Chemical Treatment.

A tank at ground level receives sulphuric acid which is conveyed to the plant by motor tank

in which case the tanks are each fitted with a vertical shaft and horizontal arm or blade mixers of cast iron, or the liquid may be effectively agitated by compressed air. As each tank deals with only one charge of liquor per day and as 20 minutes' agitation suffices for each treatment, the use of compressed air is frequently resorted to, thus avoiding the capital outlay for mechanical agitators and driving arrangement. Steam jet aspirators are largely used instead of an ordinary air compressor. The tanks illustrated are agitated by steam jet blowers having a 3 in. intake for air and a 3 in. delivery pipe. The air is distributed throughout the bottom of the tank by perforated piping, and the whole of the liquid is agitated from bottom to surface.

Settlement.

The necessary period of settlement depends upon the specific gravity of the sodium sulphate solution which results from acid treatment, also upon the temperature and upon the degree of acidity. In a

suitably arranged plant settlement should occupy not less than 12 hours for efficient results to be obtained.

In Figure 4 there are six tanks constructed in redwood of 4" thickness, each having the dimensions 10 feet square and 6 feet deep, and at total capacity of approximately 32,400 gallons. As the daily flow in this case is 22,000 gallons, it will be seen that there is sufficient tank capacity for a settlement of twelve hours.

Decantation.

The clear acid water is removed by means of a swivel arm outlet, which discharges into a special channel used only for this purpose, this liquid being conveyed to a receiving tank for further treatment.

Filter Pressing the Magma.

The magma left behind in the tank is then dealt with for the recovery of grease. It is either pumped from the tank by means of a special pulsometer pump which forces the magma into a press manufactured by Messrs. Manlove, Alliott and Co., Ltd., Nottingham, or as shown in the illustration, it is allowed to flow through cast iron pipes into a cast iron receiver or steam ram which forces it into the filter press, the pressure of steam utilised for this purpose being 50 lbs. per sq. inch. The charging is continued until the press is filled. In order to extract the grease thoroughly it is necessary to heat up the contents of the press, which is effected by means of open steam which passes along the feed pipe and into each chamber of the press. Steaming is continued at a pressure of 40 to 50 lbs. per inch until grease is no longer discharged at the filter outlet taps. The press is then opened out and the press cake removed.

Figure 4a illustrates the compressed air ram for charging filter press. In addition to the filter press there is a type of press known as an oven press, which is extensively used for oil recovery. The use of this press, however, involves much hand labour. The magma from the tanks cannot be sent directly forward to the press. It must first be discharged on to coarse filters whence it has to be removed by hand and wrapped up in canvas packages, each holding about 10 lbs. of magma. These are again packed into the press by hand. Subsequently the press is heated by steam and pressure is exerted on the packages by means of a hydraulic ram which communicates the pressure through a flat iron plate having an area slightly less than that of the press. It is desirable that this hand labour should be eliminated, hence the extended application of the filter press.

Recovery of the Oil.

The magma before pressing contains 70 per cent. of water, and the oil from the press outlets is therefore mixed with a quantity of water, which is removed in the separating tank shown in Figure 4, and the oil conveyed to a refining tank, where it is treated with about 1 % by weight of sulphuric acid, agitated, and allowed to settle when residual traces of water and certain organic substances settle out. These settlings are removed by an outlet tap and added to a further batch of scouring liquor. After clarification the oil returns to the factory, where it is utilised for the lubricating of further supplies of raw material. The oil therefore passes through the manufacturing and recovery cycles indefinitely. New supplies of oil are only purchased to make up for that portion which is not recovered in the purification process.

FIG. 4.

Under full working conditions the plant illustrated in Fig. 4 gives the following figures:—

Weekly quantities.	
Weight of soda ash used for scouring	2 tons.
Weight of soap used for scouring	1 ton = 1,456 lb. oil.
Weight of cloth scoured	43,200 lb.
Weight of oil in cloth (9%)	3,888 lb.
Quantity of scouring liquor treated per week	120,000 gallons.
Sulphuric acid used for treatment	3 tons 80% acid.
Steam used for pumping, agitation and pressing, equivalent to	30 cwt. coal.
Weight of oil produced from—	
1. Original oil in cloth	2,722 lb.
2. Soap	1,019 lb.
Total	3,741 lb.
Weight of press cake produced	4,015 lb.

(Cake contains 20% moisture and 20% oil.)
The recovered oil contains 85% of saponifiable matter.

For many years the press cake either was burned in the steam boiler furnace or disposed of as waste. Recognition is now given to the fact that this is a serious waste of valuable material, and by a solvent extraction process the oil is being extracted from the press cake, and the extracted materials disposed of as a useful fertiliser. By extracting oil from the press cake the total recovery of oil is equal to 90% of that originally contained in the cloth, together with the oil contained in the soap.

The following are analyses of cake before and after solvent extraction: —

Before extraction.		
Moisture	19·07	per cent.
Grease	21·20	,,
Ash (mineral)	41·80	,,
Organic matter insoluble in ether	17·93	,,
Nitrogen	—	
After extraction.		
Moisture	2·00	per cent.
Grease	1·05	,,
Ash (mineral)	—	
Organic matter insoluble in ether	—	
Nitrogen	4·20	,,
as NH_3	5·10	,,

Owing to its comparatively high nitrogen content it is in considerable demand as a fertiliser.

Treatment of Second Portion of Scouring Liquor.

That portion of the scouring liquor which is not sent to the recovery plant, and which represents two-thirds of the total scouring liquor, receives a separate purification treatment, to achieve one of the following objects: —

1. Purification for re-use.
2. Purification for discharge to river.

Purification for Re-use.

It has been found that the second portion of the scouring effluent can be treated in such a way as to obtain a resultant water which is quite satisfactory for further manufacturing operations.

In Fig. 6 will be seen a plant which effectively purifies this second portion. The water before treatment was found to contain soap, emulsified grease, fibrous matter and other organic solids, and sodium carbonate.

It will be observed that the necessary chemical treatment consists in neutralising the sodium carbonate with sulphuric acid, which at the same time decomposes any free soap dissolved in the water. The addition of a second reagent, *i.e.*, crude sulphate of alumina (aluminoferric), accelerates the rate of separation of the suspended solids.

The method of working the plant will now be described.

Screening.

This operation is essential, for the reasons given previously.

Pumping.

The effluent is delivered to the treatment tanks by any suitable pump. In this particular instance a direct acting piston pattern pump was utilised, and this pump was fitted with vulcanite covered steel ball valves. It was found that after six months working the vulcanite coating had swelled considerably, owing, it was concluded, to the absorption by the vulcanite of the free oil contained in the effluent. The substitution of iron ball valves proved a satisfactory remedy.

Fig. 4A.

Chemical Treatment.

A 20% solution of sulphuric acid is prepared at ground level and elevated into a overhead receiving tank. Sulphate of alumina is also dissolved to make a 20% solution, which is elevated to a similar overhead tank. These reagents are admitted to the effluent in their correct proportions by means of a suitable apportioning apparatus. It should be

stated that in order to obtain an effluent of unvarying quality, a reserve tank is provided into which the effluent flows from the scouring machines. This tank holds three hours' supply and thereby an average effluent is obtained.

Figure 7 is a drawing of the chemical system for automatic treatment. The effluent to be treated is pumped into No. 1 tank, which is fitted with a slotted outlet pipe and a float. Should there be any variation in the flow of effluent, the level of the liquid undergoes a consequent change in level which communicates itself to the float. By means of a rod the float is connected to a lever, at the opposite end of which are suspended two slots with flexible outlet connections. The slots work in separate tanks in which the sulphuric acid and alum solutions respectively are maintained at a constant level.

The widths of the dipping slots are adjustable, and when set to discharge that quantity of reagent which effects the correct chemical treatment for a given flow of effluent, it is found that automatically the reagent feed adjusts itself for any variation in flow of the effluent.

The effluent is first treated with sulphuric acid in such quantities as to reduce the alkalinity of the effluent to 4 parts per 100,000. The treated effluent enters the clearing tank, A, by means of a downtake mixing pipe, which conveys the effluent nearly to the bottom of the tank. After travelling the height of the tank, it is found that almost the whole of the precipitated grease and soap has been left behind in the tank. At this point the effluent receives the correct quantity of alum solution and enters tank B, where it undergoes the same sedimentation process as tank A. The effluent is finally filtered through a wood fibre filter of the upward flow type, and proceeds to the reserve tank in a clear, colourless and neutral or slightly alkaline condition.

Fig. 6.

Figure 8 is a photograph of the actual reagent feed apparatus.

The purified effluent has proved entirely satisfactory for the scouring and washing of woollen "pieces" and has had an extended application for steam-raising purposes. There is neither scale nor corrosion to be found in the boilers.

The solids deposited by this system accumulate in the bottom of the precipitation tanks, from whence they are periodically removed by way of an outlet valve. The sludge from No. 1 tank (resulting from the acid treatment) consists almost wholly of oil,

and is therefore sent to the filter presses for oil extraction. The recovered grease increases the percentage recovery from the woollen pieces, which in this instance reaches a total recovery of 75%, excluding the oil still remaining in the press cake.

The two purification processes are quite effective in recovering valuable materials from the waste effluent, and the process, properly carried out, is very profitable.

The acid liquor, which results from the neutralisation of the scouring liquor, is despatched to the general effluent purification plant, where it mixes with waste dye liquors and various wash waters from textile finishing operations. A portion of this acid water can be used at the plant for purification of water for re-use, instead of the sulphuric acid referred to. The cost of treatment is thereby reduced and less calcium hydrate or lime will be required at the general effluent plant for neutralising purposes. When the second portion of the scouring liquor is not required for re-use, it also is allowed to flow into the general effluent purification plant.

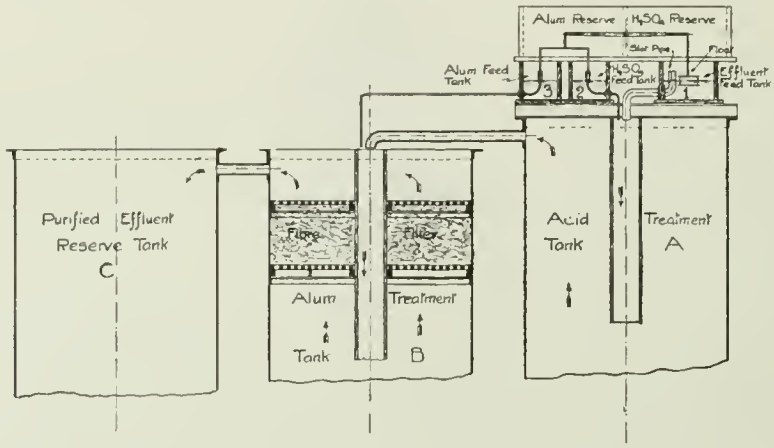


FIG. 7.

Treatment of General Effluent.

Figure 10 represents a standard type of plant for the purification of waste waters with the object of producing a non-polluting liquor for discharge into the stream. Fig. 10A is a small unit type to deal with 2000 gallons effluent per hour.

When cloth containing a mixture of wool and cotton is manufactured, waste sulphur dye liquors along with other dyestuffs may be discharged with the effluent. Ferrous sulphate is the least costly and most effective precipitant for sulphur dye effluent. These dyes depend upon sodium sulphide for their solubility in water. Treatment with ferrous sulphate converts the sodium sulphide into ferrous sulphide and the sulphur dye is precipitated. At the same time chemical treatment precipitates the other dyes contained in the effluent. The plant shown in Fig. 10 consists of chemical treatment apparatus, tapered settling tanks, and filter. It is of the continuous working type. For a given flow, the capacity of the tanks depends upon the rate of settlement of the suspended solids, which again depends upon the size and specific gravity of the particles. The temperature of the effluent has also much to do with the rate of settlement. This fact will be readily appreciated if a hot liquid containing suspended matter is observed in a glass cylinder. It will be found that during the period of cooling there is a continuous circulation in the vessel. The suspended particles are carried upwards by central convection currents and downwards at the side of the vessel by the water which is being cooled by the surrounding atmosphere.

In settlement tanks the disturbing effect of movement by convection can be minimised by causing the effluent to flow in a shallow layer over the mass of cooler water beneath. To obtain this condition of flow, the ingoing effluent should enter the tank over a sill, which should be the full width of the tank.

A baffle board should be fixed across the tank at a distance of 1 ft. from the sill, and the board should be immersed in the effluent to a depth of 4 or 6 in. By this arrangement the suspended particles will fall out of the quickly moving surface layer into the cooler and more quiescent layers underneath. When the effluent is of normal temperature the baffle board can be carried downwards in the settlement tank to a point which would not disturb the deposited solids. By this method an upward flow in the tank is promoted. The most suitable velocity can only be determined by actual trial, and it is often advantageous to carry out preliminary small-scale tests in order to determine the maximum permissible velocity and the requisite size of settlement tank. With the type of effluent under discussion, the capacity of the tanks should not be less than six times that of the hourly flow. In the illustration the tanks have a total capacity of 56,000 gallons, while the hourly flow is 8000 gallons.

The method of working may now be described.

Chemical Treatment.

Lime and aluminoferric are in this instance used as precipitants. The lime is prepared in a lime mixing vessel. A steel basket in the side of the vessel is charged with burnt lime, which is converted into milk of lime by the water in the tank. A regulated quantity of the effluent flows continuously into the lime mixer, displacing a corresponding quantity of milk of lime, which flows into the main effluent channel where it is thoroughly mixed with the effluent. A block of aluminoferric is placed in the mixing channel, where it is dissolved at the required rate. The lime neutralises the sulphuric acid (which comes from the acid treatment process of grease recovery) and a little excess lime forms with the alum a voluminous precipitate of aluminium hydroxide. This precipitate carries down with it the finer suspended matters and thereby assists in the production of an effluent free from suspended solids. The treated effluent passes in series through the four tanks. Each tank is

fitted with a horizontal baffle which prevents light floating matter from escaping. The solids are deposited in the taper portion of the tank from whence the resulting sludge is removed periodically. The taper portion of the tank is set at an angle of 60°, at which angle the sludge cannot rest upon the sides, therefore when the sludge outlet is opened the whole of the sludge flows out from the tanks.

When settling tanks having a flat bottom are utilised, the whole of the sludge cannot be removed, but only that portion which corresponds to an angle of 60° over the outlet. In such cases the tanks must be periodically emptied by hand, an unpleasant and altogether unnecessary operation. After having passed through the four tanks the effluent flows upwards through a wood fibre filter, which serves to

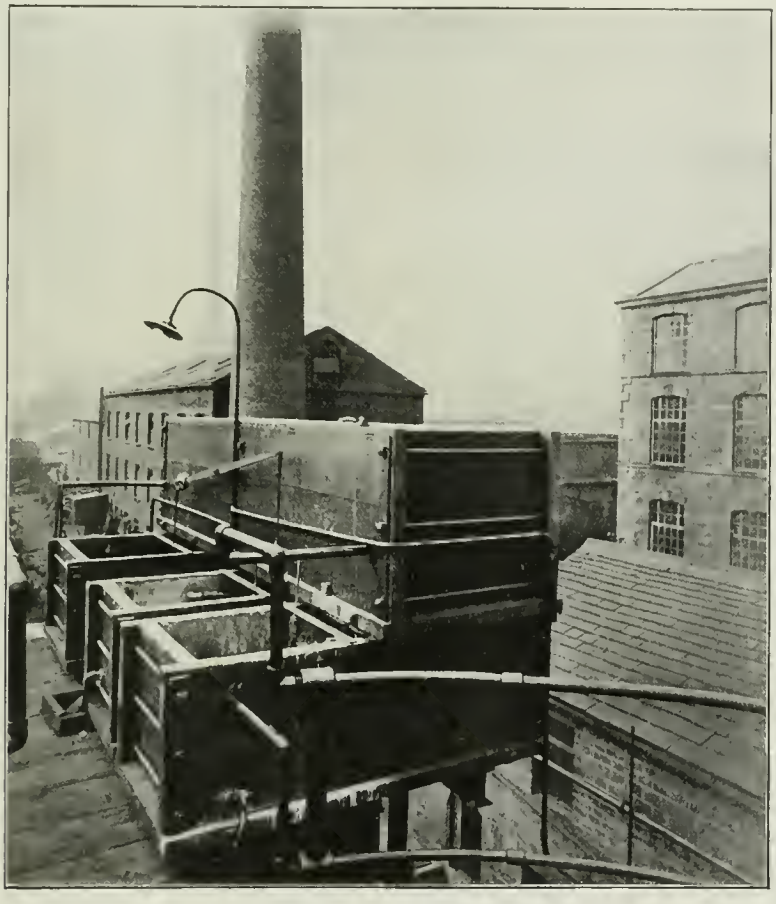


Fig. 8.

retain any light floating particles which may remain in the effluent. By this system of purification an effluent is produced which satisfactorily meets the requirements of the various rivers authorities. The effluent will be neutral or slightly alkaline, will not contain more than four parts per 100,000 suspended solids, the oxygen absorbed figure will not be high, and there is little odour or colour.

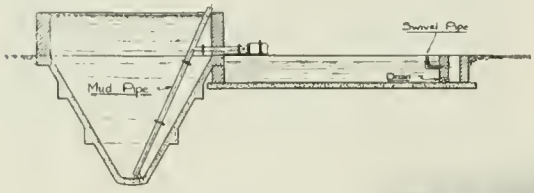
Sludge Treatment.

The sludge which accumulates in the bottom of the settlement tanks is periodically removed. In Fig. 10 there is a hydrostatic head of 4 ft. 10 in. over the sludge valve outlet, which is sufficient to promote the flow of sludge from the tank to the point of discharge. As stated previously, the whole of the sludge may be removed from the tank when the sides have an angle of 60°. The sludge is discharged on to rough filters, and may contain 95% of water. After draining a few days it will contain 70% of water, when it can be conveniently handled and carted away. The size of the mud filters is determined by the quantity of suspended solids in the chemically treated effluent. The treated effluent frequently contains 100 parts per 100,000 of effluent, in which case each 1000 gallons will produce 10 lb. of dry solids or 100 lb. of 90% sludge, which is equal to 33 lb. of 70% sludge, such as that produced after draining.

In order to promote quick draining the sludge beds should be carefully designed. Covered channels should be arranged in the bottom of the filter for the free outflow of water, and the filtering materials (ashes or stone) should be graded from the coarse material at the bottom to the fine material at the top. In effluent filters 2 in. lumps of stone or clinker are placed at the bottom to a height of 12 in., overlaid with a 6-in. depth of 1 in. material, and a final 6 in. of fine ashes such as pass through a $\frac{1}{4}$-in. mesh sieve.

In Fig. 10 the filter is also provided with a swivel arm outlet for removing the supernatant water, which otherwise would require considerable time to pass through the filter. In many installations the cost of handling and carting the sludge is considerable. There are many cases where it is less

costly to substitute filter presses for the ordinary sludge filters. This method is being adopted in a large number of plants now in course of construction. Some figures relating to an actual filter press installation may be of interest. The solids deposited in the effluent settling tanks give a total daily production of 7 tons of 90% sludge. By filter-pressing this sludge to a cake containing 50% of moisture, the total weight of sludge is reduced to 1 ton 8 cwt. There are two filter presses each having 14 recessed plates 2 ft. square. The time occupied in filling, pressing and emptying the presses is 1½ hours. One man operates the plant except when emptying the presses, when two men are required. The presses are filled and emptied ten times each day. They are charged from a compressed air ram, into which the sludge flows from the tanks, having a capacity of 350 gallons. The air pressure is 90 lb. per in. The man in charge of the presses also attends to the effluent purification plant and loads the press cake into carts for removal.



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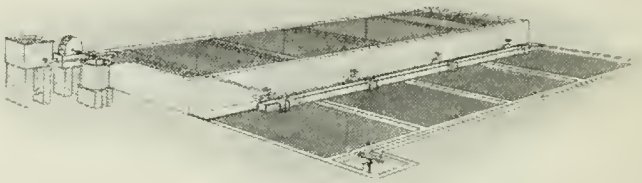
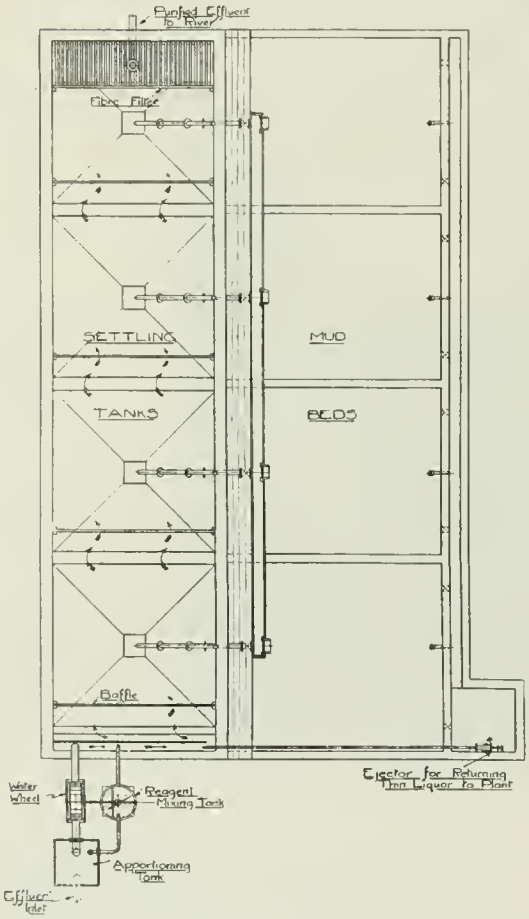


PERSPECTIVE VIEW.

FIG. 9.



— PLAN —

FIG. 10.

General Effluent Purification Plant.
Capacity 6000 galls. per hour.
For Waste, Dye waters, etc.
Patent Rotary Flock Catcher

Chambers & Hammond, Lightcliffe, Yorks.

By the three processes of effluent treatment which have now been described, namely: —

1. The recovery of grease from piece scouring liquors;

2. The treatment of the second portion of scouring liquor for re-use,

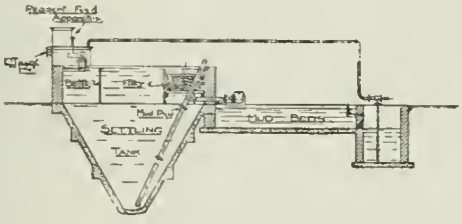
3. The treatment of dye effluent and effluent from grease recovery plant,

the whole of the effluent from a woollen cloth factory may be satisfactorily disposed of.

Figure 11 is a general diagram of a complete arrangement for effectually disposing of the various effluents from the process of cloth finishing, including the scouring, washing and dyeing of cloth.

When efficiently carried out there need be no serious river pollution, and the work may be carried out so as to give a very satisfactory return on the capital outlay involved. If a little criticism be admissible, it would be to say that if the somewhat simple operations of effluent purification were more fully understood a higher recovery efficiency would follow. This improvement can only be brought about by an improvement in the system of supervision.

There are many large textile factories whose organisation cannot be complete without the appointment of a trained chemical engineer. In many factories the value of the products recovered from such waste materials as trade effluent reaches a figure in excess of £10,000, and even in these cases there is still room for a considerably increased return as a result of scientific investigation and supervision, which could, with considerable benefit, be applied also to the purchase, selection and application of raw materials used in textile processes. In adopting this course the textile industry would be following the lead of the other staple industries.



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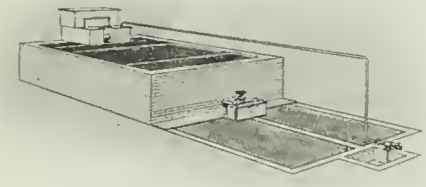

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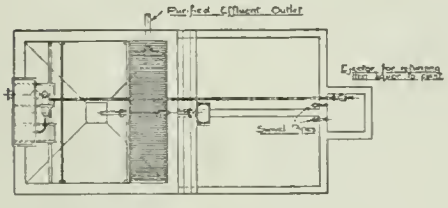

—PLAN.—

Water Purification Plant.
Capacity 2000 galls. per hour.

Chambers & Hammond, Lightcliffe, Yorks.

Patent No. 7060/1912.

FIG. 10A.

Recovery of Wool Grease and Lanoline from Wool Washings.

Another important process may now be dealt with—that of the "Recovery of Wool Greases and Lanoline from Wool Washings." Raw wool contains as impurities earthy matter, animal perspiration or suint, and yolk or wool fat.

Suint consists principally of water soluble potash salts, derived originally from the herbage on which the sheep feeds, and finally secreted from the skin as perspiration which attaches itself to the wool fibre. It is a combination of potash with a highly complex oil. Many of the volatile fatty acids, also the higher fatty acids are present in suint, including oleic and stearic acids.

Yolk or wool fat is insoluble in water but readily soluble in organic solvents such as benzene, carbon disulphide and ether. It consists chiefly of esters of the higher alcohols and fatty acids. Some glycerides are also present. About 50% of wool fat can be directly saponified in an aqueous solution of caustic potash or caustic soda.

Even in imported greasy wool the proportions and quantities of impurities vary greatly, and it is difficult to arrive at an average figure as to the actual percentages of pure wool. In the "Wool Year Book" it is stated that in 1917 Mr. H. W. Forster gave the annual consumption as 744 million pounds of greasy wool, which was stated to be equivalent to 432 million pounds of clean wool. These figures indicate that the raw wool had a percentage composition of 58% pure wool and 42% moisture and impurities. Of these impurities there are two valuable materials to be recovered, namely:—

1. Wool fat.
2. Potash.

The quantities of wool fat and potash vary considerably in raw wool. English wools contain less than imported greasy wools. Taking an average of many estimations of wool fat and potash it may be said that 100 lbs. of raw wool will produce 10% of wool grease and 5% of potash. Assuming that the average grease content of the raw wool is 10%, then, from the total raw wool consumption it will follow that 74 million pounds of wool fat are available for recovery, equal to 33,000 tons per annum.

After making an allowance for a 33% loss the figure would be 22,000 tons per annum. Figures as to the total production of wool grease are not available, but from the number of recovery plants at work such a production is to be expected. The

possibility of recovering the potash has been debated for many years. Calculating upon the figures already given, and assuming for the moment that the wool contains the equivalent of 5% of potash, there should be available for recovery, from the

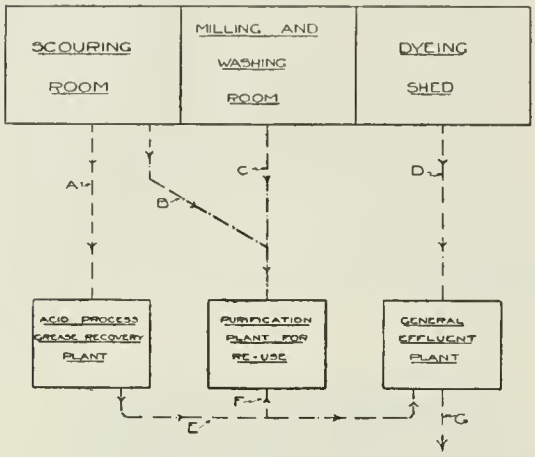

Fig. 11.

annual consumption of raw wool in the United Kingdom, 37 million pounds, or 16,500 tons of carbonate of potash.

A notable and, for a time, a successful attempt was made to recover the whole of the grease and also the potash by Mr. Walter Leach of Bradford, the process being known as the "Smith-Leach" process. Full details of this process have been given by Dr. H. Maclean Wilson in his book "Trade Waste Waters."

The spent scouring liquor was allowed to stand in large wooden tanks for the separation of sand and earthy matters. It was then evaporated in a quadruple effect evaporator to one-tenth of its original bulk. Concentration widens the difference in specific gravity between the emulsified wool fat and the aqueous solution of potash compounds. The evaporated liquor was then treated in solid basket centrifugals in which the inner layer of grease was removed by a special skimming device. The potash liquor overflowed the lip of the rotating basket and the sand remained behind.

By this process 55,000 gallons of liquid were treated weekly, producing 2 tons of wool fat, 1 ton of carbonate of potash, and about 45,000 gallons of distilled water. The plant earned a satisfactory return on capital outlay, although only working at half its rated capacity.

The initial capital outlay for the necessary plant was considerable, and no doubt with present-day fuel prices the evaporation cost would become a serious matter. Mr. Leach states that the working of this process was discontinued because of a change in the quality of wool being worked upon at the establishment in which the recovery plant had been erected.

Waste wool scouring liquor is a highly putrescible evil-smelling liquid. Even after a preliminary settlement it contains considerable quantities of suspended solids, and also free and emulsified grease ranging between 0·5% and 3·5%. The potash content varies between 0·5% and 1·5%. The nitrogen content estimated by the Kjeldahl method frequently reaches 25 parts per 100,000. At the present time, in almost every wool washing establishment, means are provided for the recovery of the wool fat. Until recent times this has been wholly effected by the acid process which has already been described. By this process the potash salts are wasted, being discharged into the nearest watercourse.

In considering the possibilities as to the recovery of the potash it was thought that a useful purpose would be served if some means could be devised whereby the wool fat could be recovered without interfering with the detergent properties of the

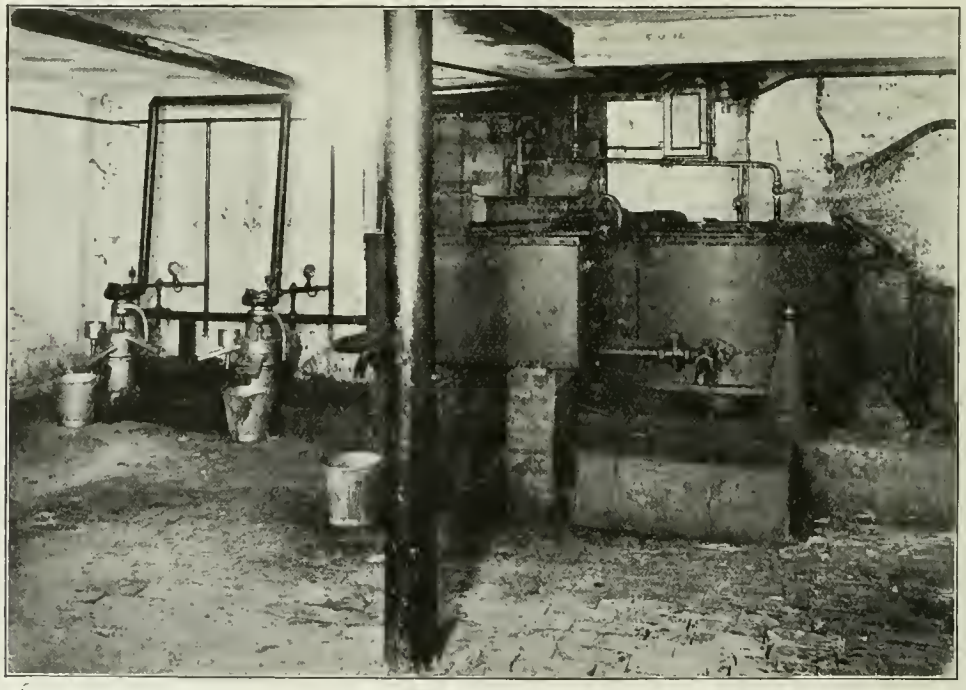

Fig. 12.

potash. To accomplish this object apparatus on a large scale was erected for the purpose of carbonating the raw wool scouring liquor, thereby converting the normal sodium carbonate into sodium bicarbonate, a non-emulsifying substance. By this process the loosely emulsified grease was separated, and after heating to remove the carbon dioxide the liquor was found to be capable of satisfactorily scouring fresh batches of wool without requiring any further addition of scouring materials.

In a series of churning experiments it was found that the loosely emulsified portions of wool fat cohered to form a solid mass of hydrated wool fat from which the anhydrous grease could be obtained. Finally, a practicable solution of the problem was arrived at by means of a centrifugal process.

of necessity of small diameter and capacity, and cannot therefore work for long before becoming filled with sand and impurities. Centrifugals of this type range between 2″ and 12″ in diameter. Large machines are manufactured up to a diameter of 42″. Such machines do not develop the centrifugal force obtainable by the small machines, but they have the advantage that they can be run for a longer period before becoming loaded with sand and dirt.

When a centrifugal machine of small diameter is adopted, it is necessary before centrifuging to remove by settlement as much sand and dirt as possible from the wool washing liquor, as such machines have little dirt-carrying capacity. The settlement, to be effective, must occupy several

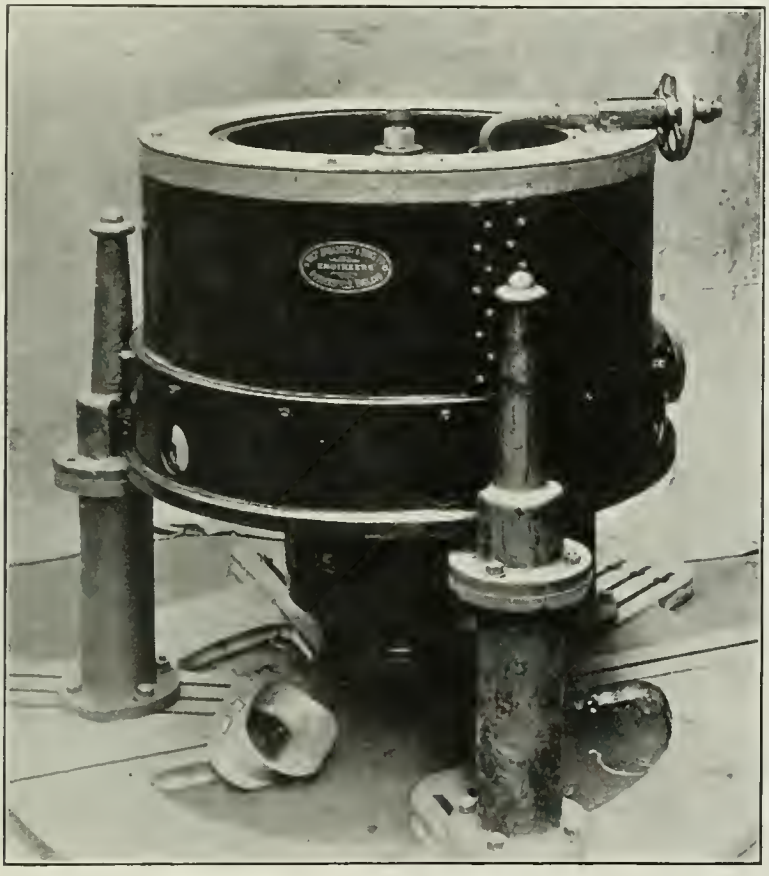

Fig. 12a.

Figure 12 is a photograph of a plant arranged for the mechanical separation of wool grease, suspended solids, and soap solution from wool scouring liquor. Under the ordinary conditions of gravity very little separation takes place between these three substances. The coarser particles of sand and earthy matter will settle out, but the grease being more or less in stable emulsion does not separate. By means of centrifugal force the effect of gravity can be greatly increased, and thereby the desired separation can be obtained. The centrifugal force is provided by means of the solid cage or imperforate basket centrifugal.

It is somewhat difficult to decide as to which type of machine to adopt for the purpose. Those centrifugals which give the greatest gravitation effect are

hours, as the magnitude of the particles of dirt ranges from that of coarse sand to a finely divided colloidal suspension. During this period of settlement decomposition in the liquid proceeds rapidly owing to the presence of nitrogenous bodies. The quality of the wool fat is affected by this decomposition, a portion of the neutral fat being split up or converted into free fatty acid, thereby considerably reducing its value as a raw material for lanoline production. It is, therefore, important that the wool washing liquor be dealt with immediately it leaves the wool washing bowl. This is rendered possible by the use of a large diameter centrifugal machine, having a large dirt holding capacity. It effectively removes the dirt in a few seconds, whereas the same result can only be

partially effected in settlement tanks after many hours. In addition, the sludge retained by the centrifugal machine is in a compact form for handling and disposal, while the difficulty of dealing with the sludge from settling tanks is well known. A portion of the grease is also continuously removed by this preliminary centrifugal, and this result, together with the removal of 95% of the suspended earthy matter, produces a liquid with which the centrifugal of narrower diameter can deal satisfactorily.

First Centrifugal Treatment.

The combination of both types of centrifugal machines is shown in Figure 12. The larger machine, manufactured by Messrs. Thos. Broadbent and Sons, Ltd., Huddersfield, has a bowl diameter of 42 inches and revolves at the rate of 1000 revs. per minute, giving a peripheral speed of 175 feet per second and developing a centrifugal force which is equal to 595 times gravity. Figure 12A is a separate photograph of this machine. The waste scouring liquor, after a preliminary screening, is heated by a closed steam coil to 180° F. It then flows into the bottom of the bowl at the rate of 300 gallons per hour, the feed being commenced when the machine has attained its normal running speed. The bowl is fitted with four vertical plates or fins which rotate the liquid at the same speed as the bowl. The thickness of the wall of liquid which is built up is determined by the diameter of the discharge lip at the top. In the machine under consideration the lip diameter is 2″ to 6″, and as the bowl diameter is 42″, it follows that the wall of liquid has a minimum thickness of 6″. A baffle ring is fixed at a distance of $1\frac{3}{4}$″ from the top of the bowl, and the baffle prevents the inner layer of wool grease escaping. The aqueous layer escapes behind this baffle and is discharged at the lip of the bowl into the casing, from which it flows by a pipe into a reserve tank for further treatment. After the centrifugal has been working for a short period the wool washing liquor separates into three vertical layers, namely:—An inner layer of wool grease; an intermediate layer of soap solution with finely emulsified wool grease; an outer layer of sand and dirt. The wall of grease gradually increases to a depth of about $\frac{3}{4}$″, when an adjustable skimming pipe with a knife edge intake is made to impinge on the surface of the grease. The momentum of the grease is sufficient to promote its passage through the skimming pipe and thence to a receiving vessel.

The dirt from the wool washing liquor is removed periodically from the bowl, the period depending upon the quantity of dirt originally contained in the liquid. On the average it may be said that this cleaning takes place every 6 or 8 hours running. On stopping the machine the liquid which falls to the bottom of the bowl is discharged into a tank containing the raw wool scouring liquor. The dirt adheres to the side of the machine and may be removed by hand in about ten minutes. The machine can be stopped, cleaned, and be at work again in twenty minutes. This particular type of machine is capable of running daily for a period of twelve hours, and in one instance for six years without any considerable charges for repairs and upkeep. When working at full speed it requires 6 h.p. The machine is of the underdriven type and works satisfactorily when directly coupled to a motor or when belt driven.

Second Centrifugal Treatment.

The liquid as it is discharged from the first machine is reheated to 180° F. and then passes forward to the high speed machines. Several types of machine may be utilised for this purpose. In Fig. 12 these machines are of the suspended bowl type and made by the Sharples Separator Company, Westchester, Philadelphia. The bowl is 4 in. diameter and 30 in. long, and rotates at about 17,000 revs. per min., with a peripheral speed of 266 ft. per sec. It develops a centrifugal force equal to about 16,000 times gravity. It is driven by a direct-coupled steam turbine working with a steam pressure of 35 to 40 lb. per sq. in. The lower end of the bowl is not secured by a bearing but is surrounded by a guide or drag bush. This guide keeps the bowl in relatively the correct position, absorbs vibration, and prevents the bowl from swinging out of the perpendicular. Owing to its high speed the bowl rotates round its centre of gravity, and even a bottom bearing would not prevent it from so doing.

The wool washing liquor is fed into the bowl at the bottom at a normal rate of flow of 100 gallons per hour. Separation takes place into two vertical layers, an inner layer of wool grease and an outer layer of soap solution. The separation and delivery of the two products is effected automatically, the wool fat being delivered at the top outlet and the soap liquor at the bottom.

Figure 13 is an illustration of another type of centrifugal machine which is being extensively used

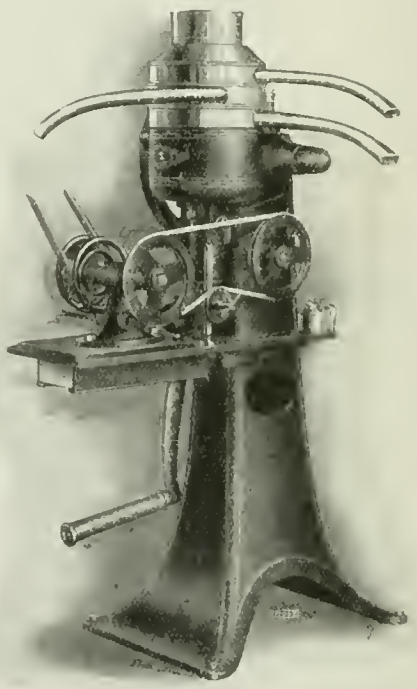

Fig. 13.

for wool grease recovery. It is of the balanced bowl type and is underdriven either by belt or steam turbine. The bowl has a diameter of 12 in. and revolves at the rate of 6000 revs. per min., giving a peripheral speed of 300 ft. per. sec., and a centrifugal force equal to 6000 times gravity. Owing to its wider diameter and larger sand carrying capacity, this machine will run for a period of 3 hrs. when dealing with a liquor which has been subjected to 12 hrs. settlement. This machine will deal with a flow of 400 to 500 galls. per hr., this larger capacity being made possible by fitting into the rotating bowl a number of light metal superimposed cones fitted to a central hollow shaft. These cones rotate with the bowl. Such an arrangement is frequently adopted in the ordinary form of centrifugal cream separator.

The purpose of these cones is to prevent the formation of eddies and to increase the length of time under centrifugal force, and to reduce the effects of viscosity and surface actions which tend to retard and diminish the separation of wool fat. In this machine the wool fat and soap solutions are automatically separated and discharged.

It will be understood that by the centrifugal process only the free or emulsified grease can be removed from the wool washing liquor. A complete separation of the wool grease is not possible with any of the machines described.

The following figures will indicate the working efficiencies of the separation process.

Results obtained by 42 in. machine (Fig. 13).

The raw wool scouring liquor treated contained:

Before centrifuging.

Free and emulsified grease	2·14	parts per cent.
Alkalinity calculated as K_2CO_3	1·12	do.
Dissolved soap	0·75	do.
Sand and other suspended matter	1·34	do.

Result after centrifuging.

Free and emulsified grease	0·86	do.
Alkalinity as K_2CO_3	1·12	do.
Dissolved soap	0·75	do.
Sand and other suspended matter	0·07	do.

Results obtained by 4 in. machine (Fig. 14).

The once centrifuged liquor contained:

Before centrifuging.

Free and emulsified grease	0·86	parts per cent.
Alkalinity as K_2CO_3	1·12	do.
Dissolved soap	0·75	do.
Sand and other suspended matter	0·07	do.

After centrifuging.

Free and emulsified grease	0·34	parts per cent.
Alkalinity as K_2CO_3	1·12	do.
Dissolved soap	0·75	do.
Sand and other suspended matter	0·0035	do.

Results obtained by 12 in. machine (Fig. 15).

Before centrifuging.

Free and emulsified grease	0·54	parts per cent.
Alkalinity as K_2CO_3	0·45	do.
Dissolved soap	0·25	do.
Sand and other suspended solids	0·34	do.

After centrifuging.

Free and emulsified grease	0·13	do.
Alkalinity as K_2CO_3	0·45	do.
Dissolved soap	0·25	do.
Sand and other suspended solids	0·034	do.

The percentage removal of grease in the above working results is as follows:

42 in. machine (Broadbent's)	60%	total grease removed.
4 in. machine (Sharples)	60%	do.
12 in. machine (Alfa-Laval)	76%	do.

Disposal of the Centrifuged Liquor.

The centrifuged liquor from which the greater portion of wool fat and the whole of the dirt has been removed has lost none of its detergent properties. The original soap added to the scouring bath is unaffected, and the liquor is further enriched by the soluble potash soaps derived from the raw wool. The liquor has a translucent appearance, and after long standing there is no aggregation of wool fat particles and no sedimentation. The liquor is therefore returned to the scouring bowl, and without any addition of soap or soda it effectively scours further batches of raw wool. Those who have had a long practical experience in wool scouring state that wool scoured by centrifuged liquor is enhanced in quality. It has been found that after long continued use the centrifuged liquor does not discolour the wool, neither is its quality effected in any way. Instances are available where the centrifuged liquor has been in circulation continuously for many months, and when fresh scouring liquors are made up exclusively from centrifuged liquor diluted with water it will be seen that a great saving is effected by this process amounting to 75% of the soap and soda previously purchased for wool scouring.

Even when no wool grease is to be recovered (as when working on non-greasy wool) the mere fact that after removal of dirt and foreign matter the scouring liquor can be used again is sufficient to yield a satisfactory return on the capital cost of the plant installed. For this latter purpose it is not necessary to use high-speed machines, as the larger diameter Broadbent's machine is quite effective.

The plant as illustrated in Fig. 12 will recover 3000 pounds of wool fat per week, and in addition effects a saving of 75% of the scouring materials which had to be used before the plant was installed. It requires the attention of one man only, the plant requires in all 8 h.p., and the value of the wool fat obtained is to-day more than twice that obtained for wool fat obtained by the older process of acid treatment. Again, the acid required by the older process of wool fat recovery, and which amounts to approximately 2 tons of acid for each ton of wool fat produced, is not required, and its cost therefore eliminated. The cost of production of wool fat by the centrifugal process is half that of the acid process, and its value twice that of the acid process.

Potash Recovery.

By the continued re-use of centrifuged liquor there is a corresponding increase in its potash concentration. Each visit to the scouring bowl results in an increase of 0.5 in the potash content of the liquor. When this concentration exceeds 3% the wool does not readily pass through the squeezing rollers of the scouring machine. The soapiness of the liquor nullifies the frictional effect necessary to take the wool into the rollers. A portion of the washing liquor must then be discarded.

This discarded liquor is the subject of special consideration at the present time, and it is definitely proposed to recover the potash contained therein. The question of potash recovery from wool-washing liquids has long been a matter for debate. The question arises—"Can a solution containing the equivalent of 3% of carbonate of potash be economically recovered?" A conservative estimate has already been given as to the quantity of potash which might be recovered from raw greasy wool imported into this country. The value of this recovered potash would be close upon one million pounds.

Previously to the introduction of this centrifugal process, the question as to evaporation cost was a serious one. By the process described owing to the continued re-use of the scouring liquor, only one quarter of the liquor would require to be evaporated. This fact has an important bearing on fuel consumption, as it reduces the quantity otherwise required by 75%. In addition, the size of the evaporating plant will be one quarter of that previously necessary, and therefore not only are the working costs reduced, but also the capital cost. Works scale evaporation tests have been carried out on a Kestner evaporator with very satisfactory results. Works scale incineration tests have also been completed, and no difficulty was experienced in producing the crude carbonate of potash. A complete evaporation plant will shortly be erected to deal with 4000 gallons per week of scouring liquor containing 3% of potash. This plant will produce 1200 lbs. per week of crude carbonate of potash.

It is very desirable that the potash should be recovered by evaporation, as by so doing there would be no discharge of polluting liquid from the wool scouring process, other than various wash waters which are quite amenable to chemical purification by the method described under the

heading "Treatment of General Effluent." A crude wool washing liquid is perhaps one of the most polluting liquids discharged from any industrial process. The following analysis, published by the courtesy of Dr. Wilson, of the West Riding of Yorkshire Rivers Board, in whose laboratory the analysis was made, is an average of a number of samples:—

Solids dried at 100° C.	2388·3	parts per 100,000.
Ash	853·0	do.
Oxygen absorbed in 4 hours at 80° F. ..	213·43	do.
Total fatty matter	834·63	do.
Unsaponifiable matter	316·33	do.
Percentage of unsaponifiable matter ..	37·86	do.
Acid required to neutralise (in terms of H_2SO_4)	385·80	do.
Total alkalinity (in terms of K_2CO_3) ..	543·30	do.
Total alkalinity (in ash)	419·7	do.
Potash (in terms of K_2CO_3)	417·7	do.
Nitrogen (from free and saline ammonia)	16·2	do.
Organic nitrogen (Kjeldahl)	33·81	do.

Even after precipitation of the wool fat and subsequent settlement and filtration (as in the method described for treatment of scouring liquor in a cloth establishment), together with a lime neutralisation treatment, the effluent is of a highly polluting character, owing chiefly to its high nitrogen content. It has been suggested that treatment on biological filters would oxidize the nitrogenous impurities and thereby render the liquid more suitable for discharge into the watercourse.

It will be evident that the treatment of waste wool washing effluent can be satisfactorily accomplished and figures are available to show that the process is being carried out in a very profitable manner. The statement may be supported by the fact that many of these installations are at work and in course of construction. In wool washing, as in many other industries, there is a growing appreciation of the fact that values have changed considerably since the year 1914, and very close attention is therefore being given to any practicable method of waste elimination.

The detergent action of Soaps is a matter of considerable scientific and technical interest at the present time. Information of great value in regard to the physical properties of soap solution is presented in the "Third Report on Colloid Chemistry," published for the Department of Scientific and Industrial Research under the heading "Colloidal Chemistry of Soap," by Professor J. W. McBain.

The following statement, taken from the above work, may be quoted:—"Krafft has quite rightly insisted that the soaps must be in solution in order that such solutions should exhibit appreciable detergent action," and again it is stated that "hot water is necessary for tallow soaps which consist largely of palmitate and stearate; as Jackson (Cantor Lecturer) has pointed out, potassium soaps are more soluble in the cold than sodium soaps."

It would appear that this statement has an important bearing upon the value of centrifuged liquor as a detergent for wool. It has been pointed out that this liquor consists of a solution of the potassium salts of various fatty acids. With an ordinary scouring soap of good quality the temperature of the scouring bath should not exceed 130° F. on fine wools. It may be stated that the more soluble the soap (provided the colloidal condition be maintained) the more effective will be the detergent action at a given temperature.

A potassium soap being more soluble than a sodium soap, in the cold, it might be supposed with some degree of probability, that wool washing can be carried out at a lower temperature by the recovered potassium soap than by any of the sodium soaps.

Scouring at a lower temperature will also tend to minimise the effect of the detergent upon the fat which is found to be an essential part of the nucleus cell of the wool fibre and which when removed leaves the fibre in a harsh condition.

Production of Lanoline from Wool Fat.

Wool fat which has been obtained from wool washings by the acid recovery process is quite unsuitable for the production of lanoline owing to its high content of free fatty acids, which in some cases reaches 60%. The British Pharmacopœia gives the following definition of lanoline:—

A yellowish, tenacious, unctuous substance.
Almost inodorous.
Melting-point about 40° C.

A solution of 0·1 grm. in 5 mm. of chloroform and 0·5 mm. of acetic anhydride when poured upon the surface of 5 mm. of H_2SO_4 in a test tube should produce a purplish brown ring, the upper layer gradually changing to green.

A solution of 0·2 grm. in 10 mm. of either should remain colourless on addition of 2 drops of phenolphthalene solution. This solution of lanoline should turn to a deep red on addition of 1 drop of normal solution of NaOH.

On heating with NaOH no ammoniacal odour should be evolved.

Ash not more than 0·3%.

Wool fat obtained by the centrifugal process, and which has been carefully washed for the removal of soluble and suspended impurities will comply with the foregoing specification. The wool fat for this purpose should be obtained from a wool washing liquor, which has not been allowed to stand for more than a few hours, as otherwise, owing to the hydrolysis of the neutral fat, the resulting product will be found to contain too high a proportion of fatty acids to meet the requirements for lanoline.

It is hoped that sufficient has been said to indicate the importance of waste recovery from the various effluents associated with the woollen textile industry. There is still scope for further investigation both in regard to the recovery of waste products and also as to the more efficient operation of the manufacturing processes involved. Careful and continued investigation and research will undoubtedly be of great benefit to the industry.

Another important trade is that of wire drawing. Centres are established in various parts of the British Isles. In the West Riding of Yorkshire large and important works are situated for the production of various grades of wire, which consists in drawing steel rods through dies which reduce the diameter to a given gauge. Before the wire can be drawn it must be cleaned. The cleaning process consists in removing the external rust (which consists of mixed oxides of iron) by means of hydrochloric acid, for which purpose the coils of wire are dipped into the hydrochloric acid. This treatment dissolves the oxides of iron, and therefore cleans the surface of the wire, which, after washing with water, is dipped into a tank containing milk of lime to neutralise residual traces of hydrochloric acid. It is also said that the lime treatment prevents subsequent oxidation. The hydrochloric acid is mixed with water in varying proportion before being used for the cleansing or "pickling" process. Under ordinary conditions the hydrochloric acid solution is diluted to a strength of sp. gr. of 1·05.

The filtered waste liquor gives on analysis the following figures:—

Ferrous chloride	18·30	parts per cent.
Hydrochloric acid	0·40	do.
Water	81·30	do.

Figure 14 is a photograph of a plant which deals with 1000 gallons of waste wire cleaning liquor per day, having the composition shown by the foregoing analysis. The process to be described consists in treating the waste liquor with sulphuric acid and subsequent distillation for the recovery of hydrochloric acid. This treatment converts the dissolved iron into ferrous sulphate which is also recovered.

The waste "pickle" is brought from the works in 10-gallon carboys, and these are emptied on to one of the filters shown. The filters are constructed in brickwork with a concrete bottom, into which suitable channels are arranged for the outflow of filtered liquid. The filters are treated throughout with a thin solution of bitumen, for protection against the slight proportion of free hydrochloric acid in the liquid.

The filtrate is collected in a stone tank, and from thence is raised by compressed air lift system into one of the larger treatment tanks. Three of these tanks are provided, each holding 1300 gallons. At this stage the chemical treatment takes place. Sulphuric acid of 80% strength is added in proportion equivalent to the equation

$$FeCl_2 + H_2SO_4 = FeSO_4 + 2HCl.$$

It is found that 1000 gallons of "pickle" require 1700 lb. of 80% sulphuric acid (B. O. V.).

In the cold there is no emission of gaseous hydrochlorie acid. The treated liquid is effectively agitated, and is then elevated as required into an overhead supply tank. From this tank a regulated quantity is continuously delivered to the distillation plant, which in this case takes the form of a cascade. It is of the usual type, and is fitted with 14 enamelled cast iron basins each 12 in. diameter. Each basin delivers to the next one by means of a deep spout. The cascade is heated by producer gas through four 1 in. Bunsen burners. The cascade is covered by obsidianite blocks and two vapour outlets are provided for the distilled vapours. It is found that from the upper basins the vapour contains but a small proportion of hydrochloric acid, but that acid of 1·11 sp. gr. is given off at the lower basins. Where desirable the strong and weak acids may be separated. The average strength of the hydrochloric acids is 1·055 sp. gr., and as this is the strength at which the acid is used in the "pickling" process, there is no necessity to separate the two distillates. The vapours from the two outlets pass into their respective condensers. The outlet pipes and condensers are of stoneware. The condensers are of the S bend type. Each condenser contains seven pipes, each 5 ft. long, in the straight portion and 3 in. diameter. Water is sprayed over the pipes for condensing purposes. The condensate flows into a stoneware receiver, and from thence the recovered hydrochloric acid runs into carboys which are returned to the acid pickling baths. Any uncondensed fumes pass into a stoneware absorption tower packed with earthenware rings and supplied with a little water. The system works on the natural draught produced by the chimney. During the period of distillation of the hydrochloric acid the boiling point is 115° C.

The liquid which passes out at the bottom of the cascade consists of a saturated solution of ferrous sulphate containing in suspension anhydrous ferrous sulphate. It is conveyed by means of a lead-lined trough into crystallising tanks constructed from stone slabs, each 9 ft. long by 4 ft. wide by 1 ft. 6 in. deep. When one of the tanks is almost filled, sufficient water is added to convert the anhydride and monosulphate into the crystalline ferrous sulphate or copperas. This solution is allowed to cool when the ferrous sulphate crystallises out. The mother liquor is drained away into a separate stone tank, and is returned by means of a steam ejector to the large chemical treatment tank. The ferrous sulphate crystals are drained and packed into bags.

Fig. 14.

The plant as illustrated is capable of dealing with 5000 gallons of waste "pickle" liquor per week, giving the following results:—

Quantity of liquor per week	5,000 gallons.
Sulphuric acid used for treatment	4 tons.
Producer gas (140 B.T.U.)	54,000 ft.
Steam for air compressor and agitating	1,100 lb.
Labour, 1 man per shift	3 men.

Production.

The plant produces

Ferrous sulphate (copperas)		5·3 tons.
Hydrochloric acid	400 carboys.	4,000 galls. of sp. gr. 1·05

The working results show a hydrochloric acid recovery efficiency of 86%. After making due allowance for depreciation, interest on capital, and other overhead charges, there remains a very satisfactory margin of profit by the working of this process.

Hitherto waste "pickle" liquor could only be disposed of at considerable cost. It is a strongly polluting liquor, and before the process described had been developed lime treatment was the only practicable method of purification. By this method large quantities of ferrous hydroxide were produced, which could not be dried except at great cost. Filter pressing was found to be unsuitable, and no use could be found for this sludgy product.

It is of interest to note that, owing to the increasing use of sulphur dyes in the textile trades, larger quantities of ferrous sulphate will be required for the purification of textile effluents. The market for the ferrous sulphate obtained from the wire cleaning effluent will most probably be conveniently found in its application to textile effluent purification.

Conclusion.

In conclusion, it is hoped that the matters which have been referred to will suffice to draw increased attention to the possibilities that exist in the exploitation of waste. Many years of intimate association with various industries accentuate the fact that there is a most profitable and interesting field for investigation and research in connection with the waste and by-products of industry. Courage, capital, knowledge and enterprise are the necessary equipment for dealing with this unchecked waste. Two industries only have been referred to. In these industries alone the value of the materials still wasted may be counted in millions sterling. Careful observation has shown that in other trades of similar magnitude large possibilities for waste prevention exist. The work is full of interest, and every successful unit of waste prevention promotes a feeling of satisfaction to its originator.

Discussion.

Before opening the meeting for discussion, Dr. Hodgson called upon Mr. H. Talbot (Hon. Sec. of the Chemical Engineering Group) to explain the origin and objects of the Group and intimate the practice of the Group in conducting discussions at its meetings.

Mr. O. Reynard, of the Bradford Wool Extracting Co., Bradford, commented on the pioneer work done by the lecturer in developing schemes of waste recovery, and in particular paid tribute to the efficacy of the screening device for separating fibre from the waste liquor. His (Mr. Reynard's) experience chiefly lay in the direction of treatment of effluents from wool scourings. He had tried the centrifugal method of extraction of wool fat advocated by Mr. Chambers, and, although qualitatively it was a pronounced success, he had only been able to secure about 40 % of the available wool fat instead of 60 % or 75 %, as mentioned by Mr. Chambers. He (Mr. Reynard) thought the explanation of the difficulty in effecting a bigger extraction was to be found in the existence in the emulsion of the wool fat two degrees of "fineness." The coarser of the two was rapidly removed and represented the 40 % extraction that he had been able to obtain on a commercial scale; the other it is not possible, with present apparatus, to separate completely, as the fat appears to approximate more to the nature of a true solution. Mr. Reynard made reference to the "frothing process" of extraction of wool fat, and also to attempts which had been made to extract the soluble potash salts by cold soaking.

Mr. E. A. Alliott, of Messrs. Manlove, Alliott and Co., Nottingham and London, made reference to the best practice in steaming the magma in filter presses for thorough extraction of the fat. He also inquired from Mr. Chambers the extent of foaming experience when evaporating the liquor for the recovery of potash salts.

Mr. Jaffe (Messrs. Richardson and Jaffe, Bradford) asked whether any steps were taken to eliminate arsenic in the acid recovered from waste pickle liquors, as presence of this element led to detrimental action on the iron in subsequent use of the acid.

Mr. Chambers, in his reply, thanked Mr. Reynard for his remarks, and pointed out that the results given in the paper had been for the most part obtained on a commercial scale at the works of Messrs. Jarmain and Sons, Kirkheaton, Huddersfield, owing to the kindness and interest of Mr. Geof. Jarmain. In particular the frothing process had been tried, but invariably led to bacterial decomposition of the wool fat unless steps were taken to sterilise or filter the air before use. The churn process had also been tried, but was only partially successful, and far less effective than the methods described in the paper. With reference to the fineness of the emulsified wool fat, he (Mr. Chambers) was of opinion that developments of the centrifugal process were easily possible, and would effect a very considerable increase in the percentage recovery.

In reply to Mr. Alliott, Mr. Chambers explained that the frothing of the potash solution was rather an advantage than the reverse, and the "Kestner" type evaporator, which had been exclusively used, worked admirably under these conditions.

Dealing with Mr. Jaffe's point, Mr. Chambers said that no trouble had yet been experienced from arsenic contamination in the recovered acid, the design of plant being such as to obviate this.

Dr. Hodgson closed the meeting by asking them to pass a very hearty vote of thanks to Mr. Chambers for his paper, which was passed with acclamation.

THE INDUSTRIAL APPLICATIONS OF ELECTRICAL PRECIPITATION METHODS.

By Dr. H. J. BUSH.

(A paper read at a Joint Meeting of the Chemical Engineering Group with the Manchester Section of the Society of Chemical Industry at the Textile Institute, Manchester, on November 4th, 1921. Mr. John Allan, Chairman of the Manchester Section of the Society in the Chair.)

In 1824, Hohlfeld, of Leipzig, suggested the removal of suspended particles from gas by means of electrical discharges. He found that if a wire hung in a bottle filled with smoke were electrified, the smoke cleared rapidly, and a deposit formed on the sides and bottom of the bottle. His suggestion seems to have been forgotten until about 25 years later, when C. F. Guitard of London again called this phenomenon to public attention, but nothing further seems to have resulted. In 1884–6 Sir Oliver Lodge again brought up the subject in a series of articles, his researches and discoveries being made independently of the earlier investigations. The object he had in view was to use electrical discharges as a means of dissipating fog and smoke. In 1885, Sir Oliver Lodge, in conjunction with A. O. Walker and W. M. Hutchings, carried out a series of experiments on lead fumes at the Dee Bank Lead Works in Wales, but the means of generating electricity at their command were not sufficient to develop the system into a technical success, and the work was abandoned. The work led to the first patents being taken out in England and other countries in 1884 to 1886. During the same period, and quite independently, Dr. Karl Moeller of Brackwede, Germany, experimented with the electrical precipitation of dust and fume, and obtained a patent in Germany in 1884. No commercial application, however, was made of this method.

During the following 20 years, beyond an occasional article calling attention to the possibilities of electrical precipitation, and a few patents on details and modifications issued, very little was done and no permanent commercial installation resulted.

In 1906, Dr. F. G. Cottrell, present Director of the United States Bureau of Mines, who was then Professor of Physical Chemistry in the University of California, had occasion to repeat the early experiments of Lodge, while studying the removal of acid mists in the contact sulphuric acid process. He became convinced of the possibilities of commercial application of electrical precipitation; and tests made at the Hercules Works of the Du Pont de Nemours Company at Pinole, California, in 1906, demonstrated this conclusively. The first commercial installation of any size was made at the plant of the Selby Smelting and Lead Company, on San Francisco Bay, for the collection of sulphuric acid fumes arising from parting kettles.

The Western Precipitation Company and the International Precipitation Company of California were organised in 1907 for the purpose of administering the patent rights of the Cottrell processes in domestic and foreign fields respectively.

In 1912, certain precipitation rights in the United States, not controlled by the Western Precipitation Company, were offered to the Smithsonian Institution, which, however, under its charter, could not conduct the business activities the offer entailed, and a separate organisation was incorporated for this purpose. This organisation, the Research Corporation, with headquarters in New York City, is now actively engaged in this work.

In England electrical precipitation has been prosecuted independently by Sir Oliver Lodge and by his sons under the name of "The Lodge Fume Company."

To render air a conductor of electricity, the negative ions must be ejected from its molecules; it therefore requires to be subjected to ionisation, which can be brought about by various agents, such as radium, ultra-violet rays of a certain wave length, Röntgen and Becquerel rays, high temperatures, chemical reaction, and, finally, electrical discharge. The rays that effect ionisation lie in the extreme ultra-violet section of the spectrum, and the line of demarcation between the ionising and non-ionising portion is sharply defined, red rays, however powerful, not having the slightest influence on the conductivity of air.

Though, generally speaking, air may be considered an excellent insulator, measurements with delicate instruments have revealed that it will always convey a small amount of electricity, and the origin of this so-called residual conductivity has occasioned much speculation and research. Tests were carried out in ordinary rooms, in places many feet below the surface of a lake, and even in mid-Atlantic to eliminate as far as possible all disturbing influences, but, though much reduced in the last case, residual conductivity still remained. Prof. McLellan of Toronto went so far as to test a sample of air enclosed in a box made of ice, and this sample certainly showed the lowest conductivity of any. Probably this small amount of conductivity always present in air is due to the small traces of radium which can be detected in almost all rocks.

Electrical precipitation on an immense scale occurs in nature, brought about by the electrical phenomena taking place in the atmosphere acting on the particles of dust and moisture suspended therein; it is only during the last 7 or 8 years that reliable measurements have been taken of the amount of electricity passing with a flash of lightning and of the length of the disruptive discharge.

C. E. I. Wilson made a series of very valuable observations at Cambridge with the use of delicate instruments installed at two places a considerable distance apart. The charged thunder cloud produces at places in its neighbourhood certain electrical effects which are measurable, and the magnitude of which varies with the distance of the cloud and the quality of the discharge taking place to earth. It was found that the average flash of lightning had a length of 10 km. and that the amount of electricity was equal to 30 coulombs. At the observation point the potential gradient was 2000 volts per metre, and it is safe to assume that in the direct line of actual discharge it was higher still. Taking the lower value, however, it follows that the actual difference of potential before the disruptive discharge occurred was no less than 20 million volts, and calculating on 30 coulombs, the energy liberated is equivalent to that of dropping a load of 600 tons through a distance of 100 metres. It is, furthermore, possible to calculate the expanse of the cloud required to accumulate the above

charge, and this is found to be an area of one square kilometre. It is surprising, in view of the enormous amount of force involved, that the damage done is comparatively so small. Fortunately it is not necessary to face such extremes in industrial electrical precipitation. The formation of these large quantities of electricity has been explained by the action of rain drops. It had been known for some time that peculiar electrical conditions existed at the foot of waterfalls when Lenard took up his researches on the electrification produced by splashing water. He found that when water drops strike on a surface and break up, the water is positively electrified and the air negatively. Further, the impurities in the water may considerably affect the phenomenon, some more than others. For instance, Lenard found he could greatly modify the electrification by adding traces of methyl violet—traces so minute that they could not be detected by the eye.

The peculiar effect of electrification on water spray can be demonstrated by the following experiments. If a jet of water is allowed to pass upwards through a central orifice of a metal dish, the spray may be so fine that practically no sound is produced by the falling particles striking the dish. If now an electrified ebonite rod is held up to the spray, the fine particles will form larger drops and produce a very distinct patter as they fall into the dish. Again, if the shadow of a jet of steam is projected onto a screen, and the orifice from which the steam is issuing approached by a wire connected with an induction coil, the shadow of the steam becomes much darker, showing that the particles have become larger.

Rain drops rarely exceed 5½ mm. in diameter, and if they grow by condensation they very quickly break up into smaller droplets, with the resultant phenomena of electrification. Now before a thunderstorm there can always be observed an upward current of air laden with moisture. As it cools the moisture separates into drops which grow larger and larger until the upward current of air will no longer support their weight. As they drop they break up into smaller particles and spray. The larger particles are positively electrified and the fine spray is electrified negatively by contact with the air. This fine spray is carried upward by the air current, and thus the two charges become separated and a rain cloud is formed with positively charged drops at the bottom and the negatively charged fine spray at the top. If the charge is sufficiently high, a spark will pass within the cloud, and the electric field is destroyed; but imagine a horizontal wind carrying away the light spray, and thus separating the two components; there will be one cloud with an entirely negative, and one with a positive charge, and provided the potential is sufficiently high a disruptive discharge will take place from either to earth. It is actually the fact that heavy rain shows positive and fine rain negative electricity. The above is only one of a number of possible explanations that have been put forward for the electrical phenomena occurring during a thunderstorm. It will be readily seen that any solid or liquid particles straying into the powerful field established between a cloud and earth will undergo electrical precipitation in a manner similar to that practised industrially.

On the whole the passage of electricity through air is like that through electrolytes, though there are some fundamental differences. The wider the air gap separating two points of different potential, the greater the quantity of electricity passing, also the current does not increase in direct proportion with the voltage. This holds good within certain limits. When the two poles of a direct current source of supply are connected across an air gap the relation between current and potential will not follow Ohm's law; after a certain voltage has been reached no increase in current can be effected by raising the voltage. A point of saturation has been reached.

To explain the above the ions present in the air gap must be regarded as the carriers of the electric current. The number of these present at any time limits the conductive capacity of the air gap, with the result that unless their number is increased by other means, no increase of current beyond a point corresponding to their carrying capacity can be brought about by raising the voltage. This holds good only up to a certain voltage varying with the size of the air gap, above which the potential difference will set up ionisation of the gas, and the current having now more ions to transport it will again increase as the voltage rises. The gas at this stage will become as good a conductor of electricity as a first-class electrolyte, *e.g.*, dilute sulphuric acid, and this condition of high conductivity is a perfectly stable one, and no-wise comparable with a discharge from a Leyden jar. In electrical precipitation of liquids and solids from gases as developed technically, a strong electrical field is established by connecting one set of electrodes to a source of high-tension potential, and the other set to earth, the air gap between the two sets of electrodes forming a link in the circuit. The electrodes are shaped to meet the particular requirements, and may either be earthed plates opposed by charged wires between which the gas to be cleaned is led, or they may be earthed pipes with charged wires suspended in them, the gas travelling through the pipes and depositing the particles on the inside of the pipes. When a solid or liquid particle carried along by the gas current comes within a high-tension electric field it is subjected to a number of diverse influences. It may suffer polarisation of its electrical charges, tending to drive it towards the charged electrode. Secondly, it will be subjected to a bombardment of ions produced in the gas by the corona discharge of the wire electrode. This will impart to the particle a charge impelling it towards the earthed electrode. Thirdly, it will be carried away from the discharge electrode by the electric wind, which is a secondary effect of the high potential impressed upon the discharge member. The finer this member, and the more projecting points it has, the greater will be the influence of this wind. The fact that with positively and negatively charged particles in the gas, all except a small percentage are deposited on one electrode only, the earthed one, is explained by the following considerations. The positively charged particle striving towards the negative electrode meets a concentrated stream of negative ions and negatively charged particles striving in the opposite direction, the probable effect of which will be that its charge is reversed by contact with them, and the particle joins the current of negative ions and particles tending towards the earthed electrode. In this movement away from the discharge electrode the particle is further aided by the electric wind referred to above. Some positive particles will nevertheless succeed in avoiding the stream of particles and ions opposing them, and thus reach the negative electrode.

It will thus be seen that the passage of electric current through the gas separating the electrodes is influenced by the degree of ionisation of the gases. It has been found that gases derived from certain metallurgical operations are so highly ionised as to offer considerable difficulties to successful electrical precipitation. The air gap, so to speak, is short-circuited, and the potential difference that can be maintained without disruptive discharge does not suffice for establishing the required electrical field. Means have been suggested for de-ionising the gas by attaching the ions to slow-moving carriers, or by bringing about a recombination of the negative and positive ions in various ways. On the other hand, electrical precipitation cannot be carried on successfully without the help of the stream of ions sent out by the discharge electrode, and the shape and dimensions of the latter have a very material effect

on the ionisation, and must be carefully adapted to suit the conditions encountered. What may answer for one gas and for one temperature may be useless for others. Another condition for successful precipitation is that the earthed electrode and its deposit shall be maintained at zero potential. If this is not done peculiar electrical disturbances take place. Such conditions have been artificially reproduced in the laboratory, and the resulting phenomena photographed.

The effect of dust deposits on the electrodes was not clearly recognised in the early days of electrical precipitation, and some of the results were very puzzling. At night a faint glow could be sometimes observed on the earthed electrode, and the dust particles, after being repelled from the charged member, would travel along the earthed electrode at a certain distance from it, instead of depositing thereon.

Corresponding conditions were reproduced in the laboratory, and a study made of the effect of a number of dielectrics on the voltage that was necessary to produce a disruptive discharge between the charged and the earthed electrode when the latter was covered with a layer of such substances.

Figure 1 shows the appearance of a normal flash-over from a thin wire or a point to a plate. Fig. 2 shows the effect of sliding a sheet of mica over an earthed metallic plate opposed by a charged point. As the mica comes within the electric field between the two, the hitherto silent discharge is replaced by a number of disruptive discharges from the charged point to the edge of the mica. A number of other dielectrics were tried, such as sulphur, glass wool, and filter paper, and in each case it was found im-

Fig. 1.

Fig. 2.

Fig. 3.

Fig. 4.

possible to maintain the same voltage as with the metallic plate alone. After exposure such dielectrics are found to have accumulated an appreciable charge. This is especially noticeable with the mica plates, which gave sparks 2 cm. long from one point to another. Fig. 3 shows the kind of discharge obtained from a point or wire to an earthed plate covered with one of the dielectrics mentioned above. Fig. 4 shows another curious effect, the effect of placing a piece of perforated paper over the earthed electrode. It is again impossible to maintain the same voltage without arcing, and the photograph shows a luminous cone round the edges of the perforation pointing to back ionisation. The glow is a much better conductor of electricity than other portions of the gas, and this decreases the distance of the air gap, and therefore the operating voltage which can be maintained consistent with smooth working conditions.

I have touched only on the fringe of the phenomena which have to be studied in applying high-tension current to the removal of particles from gases. Much is still open to discussion, but great strides have been made since Sir Oliver Lodge's and Dr. Cottrell's first tentative attempts, and operations of electrical precipitation are becoming less empirical as the results of independent investigators are sifted and correlated, and the conditions governing the precipitation of liquid or solid particles held in suspension in gaseous fluids are more definitely established.

The large installation erected for the Ministry of Munitions at Queen's Ferry in connexion with the Gaillard sulphuric acid concentrating towers is illustrated in Fig. 5; the towers were causing considerable annoyance and damage owing to quantities of sulphuric acid emitted by the stacks. Fig. 6 is a plan and sectional elevation of one compartment showing the electrodes and the manner of carrying the high-tension bus-bar supports. The plant proved to be a profitable investment.

A similar installation to this, embodying a number of improvements, was completed and ready for starting up at another Government factory when the armistice was signed.

The exit gases from the Admiralty's installation of Kessler concentrators at Holton Heath constituted not so much a nuisance as a danger to the stores of finished explosives in the sheds, and it was decided, therefore, in 1918, to instal a number of Cottrell units to eliminate the possibility of any such contamination.

Alternate scrubbers were converted into electrical precipitators, leaving always one scrubber for two Kesslers. The gases from two furnaces were drawn through one scrubber and blown into a twin set of precipitating boxes. The results achieved were very satisfactory, the capacity provided proved ample, and the escape of white acid fumes from the individual stacks was no longer seen. In fact the air issuing from the stacks could be breathed for a short while without great discomfort. The losses never having approached the magnitude of those at Queen's Ferry, the monetary return on the plant was correspondingly reduced. This plant is fully described in " Engineering," of January 28, 1921.

The installations so far mentioned are of the plate and rod type. Fig. 7 shows diagrammatically a small pipe and wire design erected at a metallurgical works for the recovery of very finely divided, precious metal dust. The refining process adopted at this plant depended for its success on a fume recovery plant, the conditions attached to which could only be met by electrical precipitation. Not only was the fume evolved highly corrosive when wet, but the constituents of the fume when moist decomposed in contact with metal work and rapidly corroded it. This was entirely overcome by treating the gases hot in the electrical field. Steelwork was used throughout, and no detriment has been noticed so far. The recovery has proved practically complete.

In this plant a motor-generator is used to convert the direct current works supply into single-phase alternating current, and to operate the synchronous switch, whereas at Queen's Ferry and Holton Heath the works alternating current supply is fed directly to synchronous motors driving the switch. The system to be adopted depends upon local conditions to some extent, as well as on considerations of cost. If a steady and ample supply of alternating current is available, synchronous motors can be used with advantage; if the supply

At the same works a precipitator has also been installed for recovering sulphuric acid fumes escaping from parting kettles. It is essentially a copy of the Holton Heath design with a number of modifications that have been found of advantage, especially in the mode of suspending the discharge electrodes and introducing the high-tension current into the box.

A very different field of application, and one that is likely to become an increasingly important one, is the use of the electrostatic method for removing

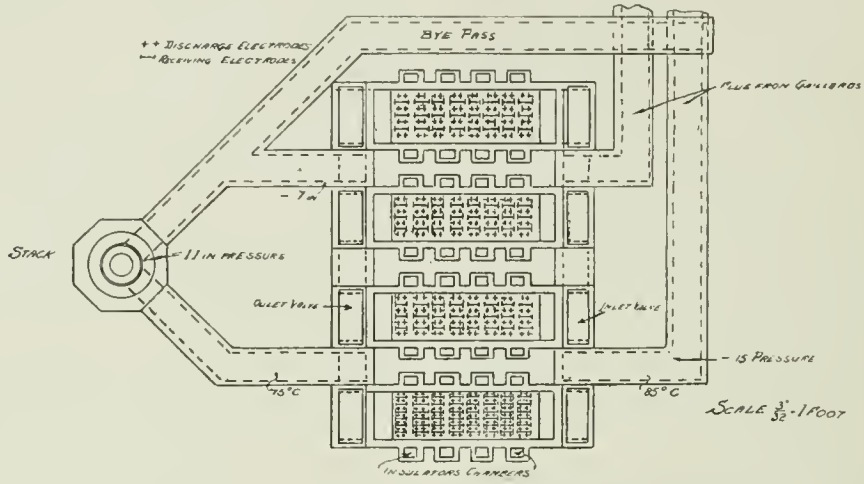

FIG. 5.

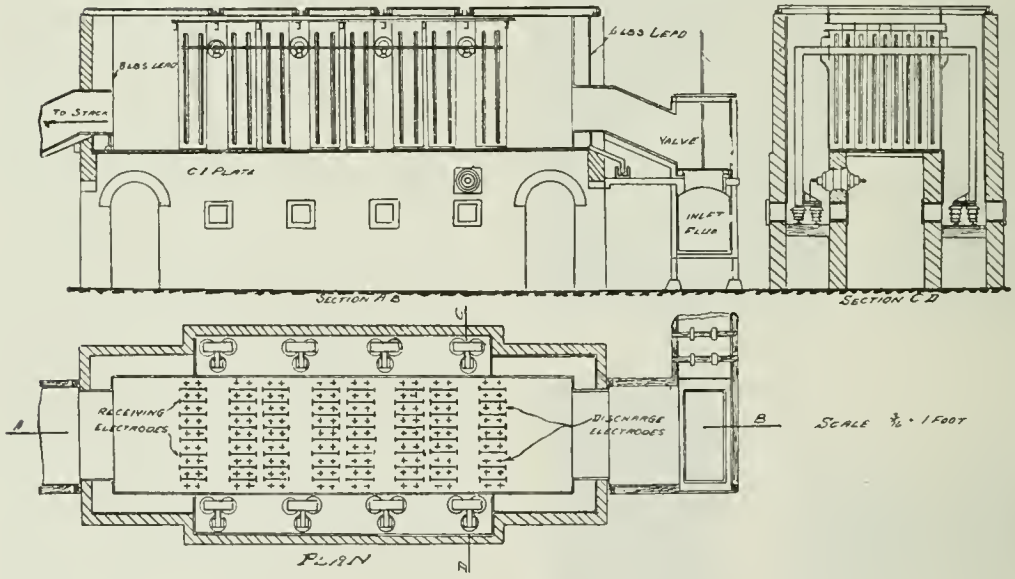

FIG. 6.

is fluctuating or only direct current is available, motor generators are called for. Special precautions must be taken to protect these machines from the oscillations that may be set up in the circuit, and which may reach considerable magnitudes. The arrangement of the bottom dust-collecting chamber and slides in this plant permits observation of the discharge electrodes at work. The corona effect along the length of the whole wire shows up very clearly, and it is soon possible to tell by its appearance whether the proper electrical conditions are being maintained in the pipes.

solid particles from blast-furnace gases. The average dust content is about 5 grams per cub. metre, rising in some cases to 15 g. per cub. m. and even more. The first large-scale installation operated in this country was designed by the Lodge Fume Co., Ltd., of Birmingham, for the Skinningrove Iron Co., Ltd. It is of the plate type, and consists of 16 compartments built in ferro-concrete, each with its own inlet and outlet valve. Ferro-concrete was adopted owing to the scarcity of steel plates. The depositing electrode plates 18 in. wide by 10 in. long, are opposed by peculiarly shaped discharge

members, there being 14,000 discharge points to each compartment. The dust adhering to the electrodes is removed by suitable rapping devices jarring both the plates and the discharge members. Blast-furnace dust appears to be particularly inclined to cling, especially if the temperature of the

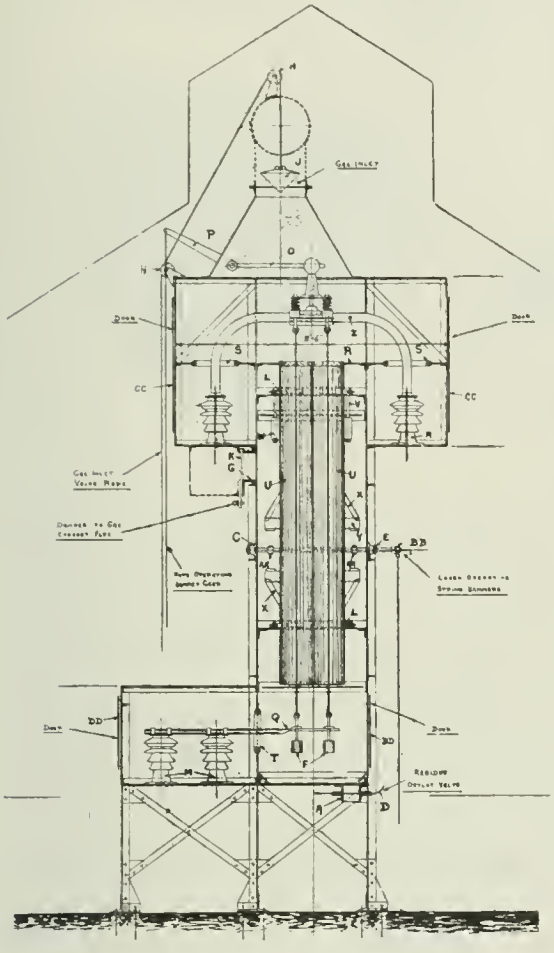

FIG. 7.

gas is allowed to fall below a point where moisture can condense on the deposit. The generating system for supplying high-tension current to this installation is of a special kind, developed by the Lodge Fume Co., and differs materially from the system followed in so-called Cottrell plants. A static transformer of the induction coil type is used in connexion with mechanical and valve rectifiers. It would seem that, while suitable for smaller installations, it has certain characteristics which make it less desirable for large units. The Skinningrove installation was decided upon in war time, with a view chiefly to secure potash, nothing beyond a rough cleaning of the gases being aimed at. About 90% of the dust is actually removed, and the rough cleaned gas is burnt under boilers and in stoves, a portion of it going through a wet washer to render it sufficiently clean for use in gas engines.* The North Lonsdale Iron Co. have a plant of the same design working on two blast furnaces on the West Coast. The dust is slightly more gritty and contains less potash than that of Skinningrove. The efficiency is higher, and the difference in the working of the boilers since they have been fired with clean gas is very pronounced. A considerable saving of coal resulted, as the clean gas made it possible to keep up steam without the use of additional coal. At the Workington branch of the United Steel Co. an electrical precipitator of the pipe and wire type is installed. It consists of six units of 64 pipes, each 9 in. in diameter, and is intended to take the gases from three blast furnaces producing hæmatite iron. A number of novel features are embodied in this plant, one of which is a mechanical gear, operated by a motor and countershaft, for hammering the pipes and simultaneously agitating the discharge wires so as to remove therefrom the deposited dust. Each compartment is self-contained, with inlet and outlet valves, rapping gear, high-tension leading-in insulator, etc., and is fed by a separate 25 kv.-a. generating unit consisting of motor generator, control panel, transformer, and potential rectifier. The whole is roofed over and means are provided for enclosing the sides should it be found desirable. The plant is completed and, but for the blast-furnaces being closed down, would now be in operation. The corona glow on the many wires as seen after dark is a wonderful sight. Incidentally, the odour of ozone escaping at the top of the pipes is overpowering.

The application of electrostatic precipitation to the cleaning of roaster gases from mechanical pyrites furnaces is a field that has been somewhat neglected both in America and in this country. In the United States isolated installations were put in as far back as 1916, but it was not until the co-operation of one of the largest acid producers was secured that results were obtained justifying the faith of the adherents of electrical precipitation. For some time past a precipitator has been in operation cleaning the gases from mechanical furnaces burning roughly 70 tons of pyrites per 24 hrs., with an average efficiency of over 98%. About 6 tons of dust is recovered per week, a large part of which would ordinarily have gone forward to block the washing towers, coolers, filters, etc. The temperature of the gases is over 500° C.; the power consumption of the cleaning plant is only 3 kw. The results of this installation were so convincing that the battery of Howard dust catchers —which are of the horizontal plate type—was replaced by electrical precipitators.

A similar installation to the above would now be working in this country if the trade depression had not made it imperative to suspend all plant construction work at the establishment in question. The materials are, however, all on the site, and it is hoped to start the erection shortly.

While the efforts of those engaged in electrical precipitation here and in the United States were directed during the war mainly to the recovery of valuable metal-bearing dust from smelting works, the Germans concentrated on the pyrites gas problem as soon as war broke out, and it must be confessed their labours were eminently successful. The dust-laden pyrites gases were found to be particularly well conditioned for treatment in the electrical field, and present none of the difficulties that had to be dealt with in the case of blast-furnace gases. No trouble was encountered in removing the deposited dust from the electrodes, and after one or two setbacks the insulation question was solved in a very satisfactory way. The design is of the plate and wire type, free from unnecessary complications, and as inexpensive as can reasonably be expected. The general arrangement of such an installation, with the generating set, control gear, and precipitator, is shown in Fig. 8. To guard against dust passing through and giving rise to stoppages in case of current failure, a system of automatic control and alarm gear was used, whereby if

* The installation is described in a paper read at the 1920 meeting of the Iron and Steel Institute in Cardiff, and a supplementary paper by the members of the Skinningrove staff was submitted at a meeting of the Cleveland Institute of Engineers, held at Middlesbrough on Feb. 7, 1921.

disturbance occurred in any one precipitating compartment an automatic cut-out would interrupt the current supply, close a valve on the gas inlet, switch on a red pilot light, and sound an admonishing hooter. These precautions were found in practice to be exaggerated, as the operation proceeded uniformly and smoothly with practically no interruption except that required for removing the accumulated dust. This entails switching off the high-tension current and changing over to another compartment. Accordingly these controls were subsequently simplified, and instead of always having one compartment idle ready for use, twin compartments are used, of such a capacity that one can handle all the gas at a reduced efficiency during the short time that the other is off for cleaning. More than 20 plants were installed during the war for cleaning the roaster gases for oleum works, among them being those of Nobel, Griesheim, Leverkusen, Walter Feld, Uetikon, and Von Heyden.

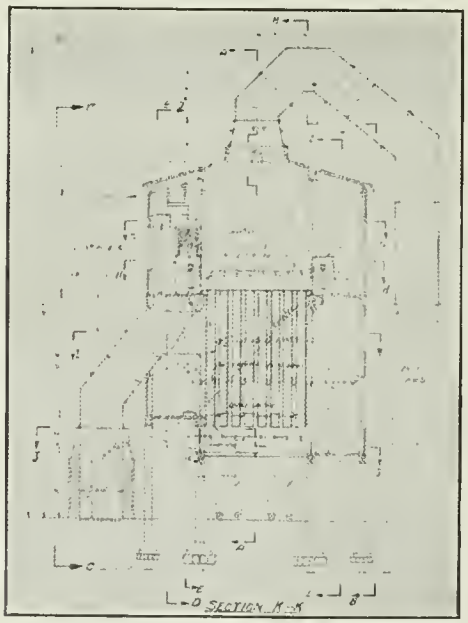

Fig. 9.

Since the war results are to hand of electrical precipitators working on mechanical roasters of lead chamber plants, and they show that there also appreciable economies can be effected thereby. In addition to the advantage of being able to produce acid of "brimstone quality" from pyrites, there is no cleaning out of flues, no blocking of Glover and Gay-Lussac towers, no silting up of coolers, tanks, and pumping machinery, and no handling of accumulated sludge with its attendant expense of labour and loss of acid—in fact the operation of the whole plant is rendered more continuous and therefore efficient. In one instance a saving of labour of 228 hours a month, and in 78% acid of 7 tons a month was recorded. A high dust content of the roaster gases is generally considered to mean a high nitre consumption, and this has been fully substantiated by the electrical precipitators. In the case of one chamber plant the adoption of electrical precipitation produced a reduction in the nitre consumption of no less than 30%, and at a new type of tower plant it was only after this had been installed that both the output and the nitre consumption were as stipulated.

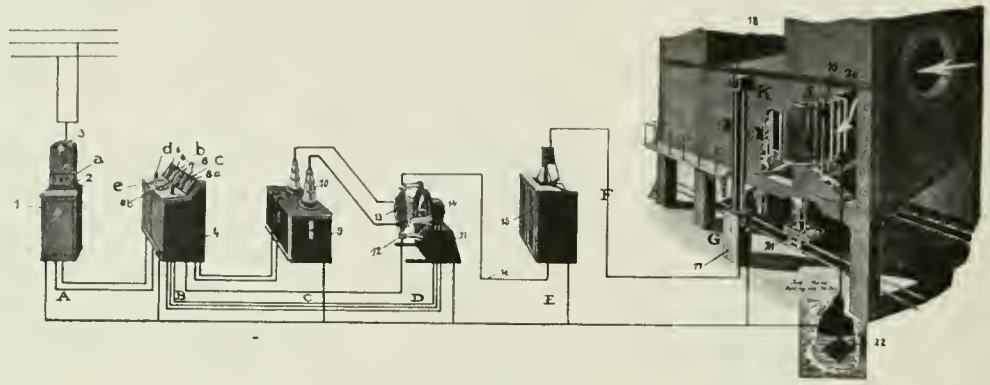

Fig. 8.

Electrical precipitators are now sold in Germany as a standard part of a mechanical furnace installation. There are a number of other installations in Germany, mostly of the pipe and wire type, working on lead smelter fumes, tin furnace fumes, alumina calcining furnace gases, and waste gases from evaporators of sulphate liquors from cellulose works. An interesting departure is a recently started plant for treating the gases from coal briquetting furnaces, and at the large Leuna works belonging to the dyestuff group a plant is, I believe, now removing the obnoxious dust from the stack gases of the boiler installation.

In Japan the electrostatic recovery process, though first introduced only in 1916, has made rapid progress, there being more than eight installations at work with an aggregate of over 2000 pipes. The Ashio copper smelter of the Furukawa Mining Co. has 640 precipitating pipes, in which from 6 to 10 tons of dust is recovered per 24 hrs., containing over 1% of copper and 30% of arsenic, which otherwise would escape into the atmosphere. The fumes are derived from McDougall roasters, blast-furnaces, and copper converters.

The erection of the Naoshima smelter, belonging to the Mitsubishi Mining Co., was licensed by the Government only on condition that the fumes from the roasters and converters were treated electrostatically before passing out to atmosphere; 1½ tons of dust is collected per 24 hrs., containing over 2% of copper, 22% of lead, 13% of arsenic, and traces of silver and gold. Fig. 9 shows a section through the precipitator at the copper smelter Ikuno, belonging to the same company.

The conditions in the smelting industry in America are, generally speaking, widely different from ours, both as regards capacity of plant and smelting practice. For instance, at the rectifier house for the Murray Smelter at Garfield there are

nine 25-kv.-a. motor generator sets installed, each driving its mechanical potential rectifier and supplying current to the primary of a high-tension transformer. The United Verde copper smelter has 3600 precipitation pipes to clean the gases from its roasters, blast-furnaces, and converters, current being supplied from 12—25 kv.-a. motor generators.

At another plant comprising seven rotary cement kilns there are five sections of 120 pipes each, in which daily 25 tons of dust can be recovered.

The Research Corporation of New York have invaded quite new fields by some of their later installations which were erected, not for the purpose of reducing dust losses on smelting operations, but solely for cleaning air. For several years during the war the air from workrooms at an American small-arms factory, where carding, buffing, and grinding wheels were operating, was cleaned by electrical precipitation. A volume of 50,000 cub. ft. of air at 70° F. was treated, and several cartloads of dust per week were removed from it. Incidentally, researches made during the war have shown that after passing air through an electrical precipitator, it is not only free from dust particles, but sterilised as well. This is due to the removal of the solid particles which carry bacteria and to the toxic effect of the ozone and oxides of nitrogen produced by the corona discharge.

At the Stace Mining Co., electrical precipitation was resorted to after cyclones, bag filters, and scrubbers had been tried without success. The problem was to remove the fine slate dust arising from the crushing plant where slate rock was passed through rollers for the preparation of slate dust for roofing material. Over 100,000 cub. ft. of air is cleaned per minute, and several tons of material are recovered per week.

An interesting installation is now under construction for cleaning the air from a works where large numbers of piston rings for gas engines are cut and ground. The precipitator will replace a bag filter, which was found unsuitable owing to the cloth cutting, the difficulty of keeping the bags clean, and the excessive power consumption required. A flue type of precipitator will be installed, having a cross-section of 14×10 ft., and a capacity of 25,000 cub. ft. of air per minute. In another case the air from tumbling barrels in an iron foundry, carrying sand and iron dust in suspension, is to be cleaned electrically, a cyclone installation having proved incapable of retaining the fine dust. On the plains outside the mining town of Pachuca in Mexico there are many thousands of tons of tailings which have been carried down by the river from the numerous milling and concentrating plants operating in that town. Several attempts have been made in years gone by to recover the metallic values from these accumulated residues, without achieving much success until chloridising volatilisation coupled with recovery of the products by electrical precipitation was suggested. A unit of 3×64 pipes is in operation there in connexion with a rotary furnace resembling a cement kiln, with a capacity for treating 100 tons of residues per 24 hrs. The pipes are of wood, rendered suitably conductive, and the discharge wires are lead-covered. The material recovered contains gold, silver, mercury, and lead.

At Mexico City an installation is doing good work at a silver refinery on the gases from refining and cupelling furnaces and desilverising kettles. Mapimi, Durango has a good-sized plant recovering over 10 tons a day from the waste gases of Huntington-Heberlein sintering pots, Godfrey roasters, and blast-furnaces. The dust has a high arsenic content, besides containing lead and antimony.

South America too has a number of installations. At the Aramayo Francke Mines of Bolivia, a plant has been installed in connexion with converters and roasting furnaces, the recovered dust containing chiefly bismuth. Installations of considerable magnitude are also being put up by the Braden Copper Co. of Chile, and the Cerro de Pasco smelter; the latter recovered during one month no less than 30 tons per day, according to information given by the International Precipitation Co.

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Discussion.

Mr. J. A. Reavell asked as to the possible application of the process to such instances as the recovery of powder in a dried milk factory. In such cases a certain amount of ozone would probably be generated which would possibly contaminate the milk and give it a distinct flavour. Also, was there any chance of an explosion occurring?

Mr. Weil asked whether there was any possibility of selectively precipitating particles according to size and what was the upper limit of the amount of dust in the gases treated. In the American process for making zinc oxide a very large amount of dust was blown over with the furnace gases. This dust usually contained particles much heavier and larger than the best qualities of oxide, and these were usually precipitated in a preliminary chamber before the pigment oxide was removed.

Mr. Drummond Paton said that in the case of the German plants the entire structure was insulated from the earth. In all static conditions the supply of current must be from the earth. He thought if the deposit that arose on different poles was analysed a selective action would be observed. Air was practically stable, and he believed the stability of any material was simply a function of the coulomb-static capacity of its individual elements. The same applied to metals either in the vapour, liquid, or solid state, and the segregation of unstable alloys under rise and fall of temperature was simply the reciprocal of the internal Chatelier effect which must take place in a composite metal or material. Eutectic and stable alloys, if considered in relation to the positive-negative capacity of their elements, taking calcium as one end of the series and fluorine as the other

(see G. Fore, "Electrolytic Separation of Metals," 1890), could be better understood than by supposed chemical affinities, and he had suggested this means to the Admiralty as a basis of defining stable alloys, *i.e.*, the blending of materials in relation to the static capacity, and by blending in functions of that capacity a stable result would result. Therefore, air, if an analysis were made on a basis of static capacity, would be practically a static material or balanced charge.

Mr. Drayton asked if the specific inductive capacity or conductivity of the chief constituents of a dust had any effect on the ease with which it could be precipitated. If the polarity of the electrode was correct the pipes to earth were positive and the wires were negative. When the polarity was negative the wire looked like a luminous string of beads. When the polarity was wrong the wire was surrounded by a thick violet glow.

Dr. Craig mentioned the successful application of the process to the fumes from the Selby Smelting Works.

Mr. Holgate said that he had to deal with gases from a blast furnace which contained 10—15 g. of dust per cubic metre, and which also were emitted at 400°—600° C. The cleaning of these gases presented a very difficult problem. The dry cleaning, or bag filtration, process did not seem to be applicable to his particular case. Two installations of the Cottrell process had been erected in America for dealing with gases such as he had to deal with, and these appeared to be very successful. At the plants he had visited in America, where the Cottrell process had been adopted, there had been a good deal of difficulty at first through the insulators having given way and the tubes having warped owing to the high temperature of the gases, but these difficulties had been overcome. In America tubes of about 6 in. diameter were recommended instead of 9 in. By that means it was claimed to clean the gases with a voltage of only about 50,000 instead of 70,000. The cost of an installation of one unit to deal with one million cubic feet per hour in 1920 had been estimated to be, roughly, $100,000. The electrical precipitation process appeared to be preferred to the steel wool filtration processes. He believed that in the Cottrell process as worked in America the gases were passed upwards through the tubes and not downwards.

Dr. Bush, in reply, said that one of the greatest difficulties they had had to contend with was that it had been found up to date practically impossible to standardise plant. Every new plant had to be specially adapated to existing circumstances. In some cases 99% efficiency was required, whilst in others 60—70% was sufficient. All such factors obviously affected the cost of a plant. It was entirely a question of the air volume treated per foot run of electrode. There was an installation at work in the United States for drying lemon juice and orange juice and also skimmed milk. He believed that no deleterious effect had been noticed with regard to the taste. The danger of explosion had been tested at San Francisco in the case of sugar dust produced from a dryer, and the difficulty had been got over in a simple manner which prevented explosion. The proportion of dust which it was possible to remove from the gases depended on the efficiency of the plant. The concentration of zinc oxide given off by evaporation processes was apt to be rather high. Although there was no difficulty from the precipitation point of view, especially with the plate type of precipitator, which had a large depositing surface, if a pipe type was used the deposit grew so rapidly that the pipes had to be cleaned out at very frequent intervals. The positive electrodes and the entire steel structure were earthed, and therefore at zero potential. He presumed the gases referred to by Mr. Holgate were ferro-manganese gases. There was no reason why such gases should not be treated successfully, although it could not be done at a temperature at which the steelwork would suffer, but there was no objection to cooling the gases and precipitating in the usual way. He did not think the Kling-Weidlein steel mattress filter would be a very serious competitor to electrical precipitation. It started up very well, but the superimposed layers of steel wool very soon got choked up, and then the filtering action stopped and they had to be cleaned. Special shaking apparatus had had to be introduced, but on the whole it had not proved very successful. The actual precipitation efficiency was not affected by the size of the pipe. It was, of course, an advantage to use a smaller pipe because of not having to deal with such a high potential. In a 12-inch pipe the actual effective voltage, measured by the spark-gap meter, was in the neighbourhood of 50,000. In this country they would probably standardise on 9-inch tubes. The smaller the tube the smaller was the depositing surface and the less the deposit would be allowed to grow. If the deposit grew beyond a certain point there was an arc from the wire. The question of up-draught as against down-draught was one on which there were differing opinions. One advantage in up-draught was that if a lot of material were deposited in the bottom of the hopper there was no danger of it being carried away by the clean gas as might conceivably occur with down-draught. On the other hand, a great feature was a good distribution of the gases in the pipes. If the gases travelled downwards, and one pipe tended to take more gas than its due share, it would get hotter than the rest and thus act as a brake. Installations both of the down-draught and the up-draught type were being put down according to circumstances. When his (the speaker's) firm had introduced the business into this country one of their first aims was to make themselves independent of foreign machinery. Admittedly the Queen's Ferry plant had American machinery, but that was simply a question of saving time. All the plants that were now being put up in this country were supplied with machinery and material made entirely in this country. Excellent porcelain insulators were also made in this country. There were quite a number of installations in this country. Besides the Workington and the Skinningrove plant there were the North Lonsdale and the Sheepbridge plant, and another one at the metallurgical works he had already mentioned, while there was a plant now being erected at a tin smelters of 10 units of 48 pipes each which would handle something like 80,000 cub. ft. a minute. The last-named plant would be made of British material throughout.

THE CLAUDE SYNTHETIC AMMONIA PROCESS AND PLANT.

By J. H. WEST, A.C.G.I., A.M.Inst.C.E.

(*A paper read at a Joint Meeting of the Chemical Engineering Group of the Society of Chemical Industry with the Institution of Mechanical Engineers in the rooms of the Institution of Electrical Engineers, London, on November 22nd, 1921. Mr. J. Arthur Reavell, M.I.M.E., M.I.Chem.E., Chairman of the Chemical Engineering Group, in the Chair.*)

THE CHAIRMAN said that the paper they were going to have from Mr. West was on a subject of great importance, and the happenings of the great war had made it of still greater importance. Mr. West had an unique knowledge of this process, and without wasting further time he would call on him to read the paper.

The Claude synthetic-ammonia process has this in common with the older Haber process, that it consists in effecting the direct combination of hydrogen with nitrogen under pressure and in presence of a catalyst to form ammonia; the predominant difference between the two processes lies in the much higher pressure used by Claude. This difference may seem at first sight rather an unimportant one, but it will presently be seen that, on the contrary, it is a most far-reaching and fundamental one, which entirely modifies the working conditions of the reaction.

In order to understand the primary considerations which led Claude to adopt pressures which had hitherto been undreamt of in industrial working, it is necessary to examine the conditions of equilibrium between nitrogen, hydrogen, and ammonia under various pressures and temperatures. Fig. 1 was prepared by Claude from his own experimental data, and shows very clearly that, for a given temperature, the proportion of nitrogen and hydrogen remaining combined under conditions of equilibrium increases rapidly with rise of pressure, and that the lower the temperature the higher is the proportion of combination.

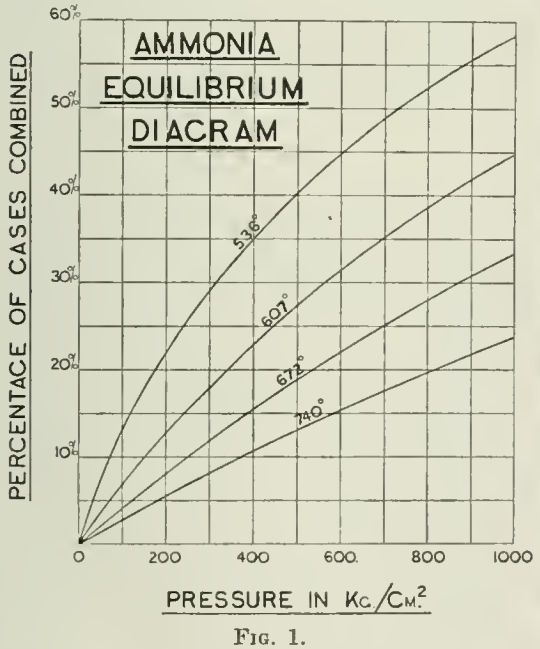


FIG. 1.

So far, the conditions for maximum combination would appear to be high pressure and low temperature, but in practice another consideration comes in, namely, speed of reaction. The lower the temperature the longer does it take to reach equilibrium, and in order to have apparatus of a reasonable size the reaction must not be too slow. Hence for practical purposes a compromise has to be made as regards the working temperature so as to get a reasonable speed of reaction combined with a sufficiently high proportion of combination of the gases. The useful range of working temperatures is found to lie between 500° and 700° C.

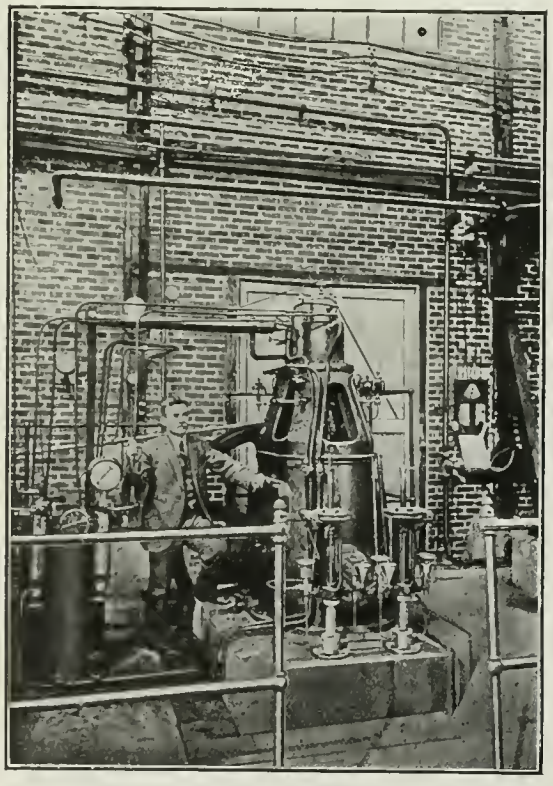


FIG. 2.

Claude finds it best to work at about 600° C., and he uses a pressure of 900 atmospheres, or nearly 6 tons per sq. in., as against the 200 atmospheres used by Haber.

The hydrogen and nitrogen, mixed in the proper proportions, are first compressed to 100 atmospheres in an ordinary three- or four-stage compressor, passed through separating bottles where any condensed moisture and oil from the compressor are drained off, and thence are led to the "super-compressor."

This is an exceedingly simple two-stage machine, capable of dealing with enough gas to make 5 tons of liquid ammonia per 24 hours, *i.e.*, a duty of 426 cb. ft. per minute.

Figure 2 shows the compressor with M. Claude standing beside it. One of these machines has been running in France for some nine months past, and has given no trouble; in fact there are few compressors for 100 atmospheres which run so sweetly and silently, and with such freedom from vibration.

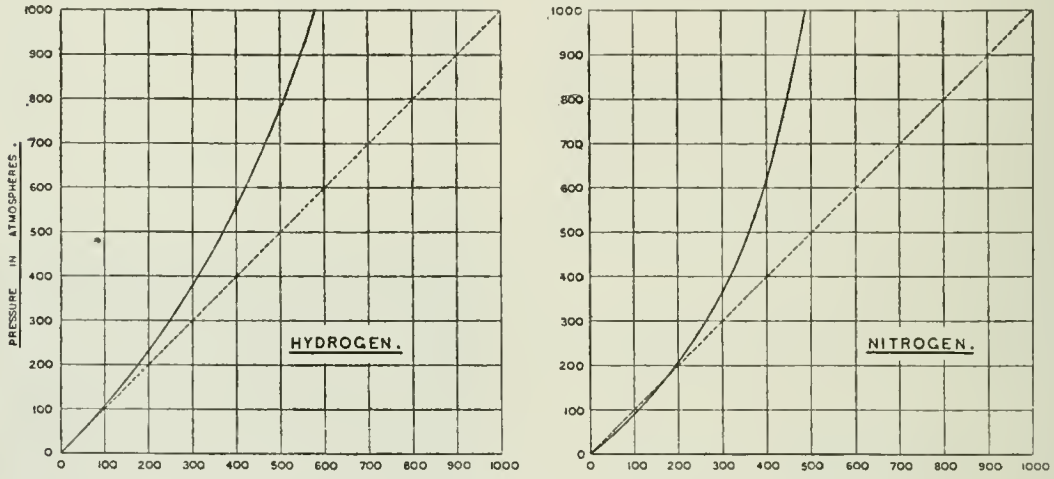
With regard to the power consumed in compressing to these super-pressures, and bearing in mind that the power varies as the logarithm of the pressure difference between intake and delivery, it will be evident that the power required to compress from 100 to 1000 atmospheres will be half that required to compress from 1 to 100 atmospheres, since the logarithm of 100 is 2 and that of 1000 is 3.

Attention may be called at this point to the very considerable departure from the gas laws of Boyle and Charles which are met with under these conditions. The curves in Fig. 3 show the volumes of nitrogen and hydrogen respectively which can be compressed into unit volume at various pressures, the dotted lines showing the corresponding volumes for an ideal gas. The expansion with temperature has not been determined above 200° C., but at 900 atmospheres constant pressure, a volume of hydrogen represented by 1000 when measured at 0° C. becomes 1449 at 200° C., whereas according to Charles' law it should become 1733.

The first result or consequence of working at super-pressures is that the pipework becomes very small. The pipes carrying the mixed nitrogen and hydrogen for a plant making 5 tons of ammonia per day are only 8 mm. bore, and the difficulty of making gas-tight joints and valves is thereby enormously reduced.

It has already been seen that the proportion of combination is much higher at 900 atmospheres than at 200, viz., about 42 instead of 13 per cent., and this has some very important consequences. The formation of ammonia is an exothermic reaction producing 717·6 calories per kg. of ammonia formed. When the proportion of combination is low, as it is at 200 atmospheres, the heat produced by the reaction is not sufficient to maintain the catalyst vessel and the incoming gases at the proper temperature unless the losses from radiation are reduced to a minimum by diminishing the radiating surfaces as much as possible. This fact compelled the Germans to devise enormous catalyst vessels, not simply for the sake of having large units, but in order to minimise loss of heat by radiation, and the preliminary heating up is done by injecting air and burning part of the hydrogen. The great cost of manufacture of these huge vessels, the difficulty of handling them, and the time lost in making the joints, can easily be realised.

Now at 900 atmospheres the conditions are quite different. The heat produced by the reaction then becomes sufficient to make it auto-thermic, so that no external heat need be supplied once the reaction has started. The reaction starts and stops quite suddenly; it does not begin gradually and build up, but starts as soon as the temperature is high enough, and goes on until for any reason the temperature falls too low or the activity of the catalyst becomes too feeble, when it stops with equal suddenness.



VOLUMES OF FREE GAS COMPOUND INTO UNIT SPACE AT VARIOUS PRESSURES.

Fig. 3.

Under Claude's conditions, then, quite small catalyst vessels can be used, which can be heated initially by external electric circuits, and after that the problem is to get rid of the excess heat. The usual way of expressing the relative efficiency of catalytic reactions is in terms of grams of product produced per hour in each litre of volume occupied by the catalyst material. In the case of the Haber process this figure is about 350—400 grams, and in Claude's process it ranges from 5600 to 6700 grm.

Figure 4 shows to the same scale the Haber catalyst vessel and the Claude catalyst tubes for a production of 20 tons of ammonia per day. The respective weights in the two cases are approximately 74½ tons and 11¼ tons.

Furthermore, in the Haber process, owing to the necessity for circulating the gas round and round over the catalyst, it is essential to have very large

and complicated heat interchangers, in order that after each passage over the catalyst the gas may be cooled sufficiently to remove the ammonia and then be heated again to reaction temperature. The time required after a stoppage to get the whole Haber system heated is three days, whereas, starting all cold, the Claude plant will begin to produce ammonia in 4—5 hours.

In the Claude plant there are four catalyst tubes, the first two of which are parallel, and the other two in series with the first two and with each other.

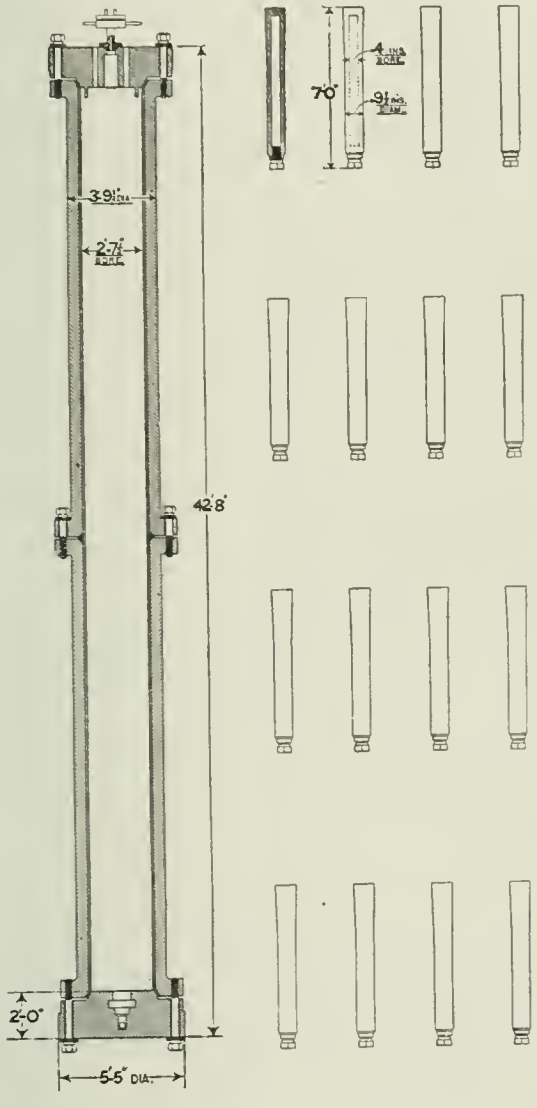


Fig. 4.

A simple calculation shows that with such a system of four tubes, if a is the fraction of the gases which combine to form ammonia in each tube, the total combination in the system will be $a(3-3a+a^2)$, and working this out for $a=0{\cdot}42$, the total combination is found to be just over 80 per cent. On actual tests the observed proportion of combination has been found to be from 81 to 83 per cent.

Now as to the actual construction of catalyst vessels to withstand the conditions of ammonia synthesis, that is to say a temperature of 600° C., a high pressure, and possible attack by the hydrogen. The Haber people first of all made an inner vessel of very pure iron and surrounded it with a layer of nitrogen under slightly higher pressure than that used inside, thus hoping to protect the outer vessel from the action of the gases. This was not a success, and they now use an outer vessel of high-tensile steel, probably tungsten steel, and protect it with a lining of pure iron. Since this lining is permeable to hydrogen, the outer vessel is perforated all over with small holes, so that any hydrogen passing through the lining can escape and not set up a pressure between the lining and outer vessel.

With the small Claude tubes it is possible to do away with all lining, and use a special nickel-chromium alloy. Some of Claude's tubes have been made by a French firm of steel makers, and some by Messrs. Vickers, of Sheffield, who had independently worked out alloys of this type. Messrs. Vickers' metal is known as "Vicronic," and it has very remarkable heat-resisting properties.

The tubes are cast solid and bored out. The head or plug is secured to the tube by an interrupted thread exactly like the breech mechanism of a gun, thus enabling the joint to be made very rapidly. The joint is made by a thin copper washer. The screwing up is done by means of a wormwheel attached to the head, rotated by two worms geared to a crank handle. The inner tube of thin sheet iron, which contains the catalyst material, is attached to the head, and it is quite easy to remove an inner tube and replace it by another containing fresh catalyst material in 7 or 8 minutes. Compare this with the time which would be required to unbolt the huge end plate of the Haber vessel, replace the catalyst, re-make the big joint, and screw up again.

Before leaving the subject of the catalyst tubes the method adopted for testing them may be of interest. It is the same as that used for testing ordinary gas cylinders. The tube to be tested is placed in a jacket filled with water and provided with a graduated tube. The test pressure is 10 tons per sq. inch, and at this pressure the expansion of the tube amounts to about 50 c.c. The method is extremely sensitive, and shows that the strict proportionality between stress and strain, demanded by Hooke's law, does not in fact exist, a fact which is now becoming generally recognised. Far below the elastic limit, and in fact all the way up the load curve slight permanent stretching takes place. It is interesting to note that at a pressure of 10 tons per sq. inch water is compressible to the extent of 6%, and that about a litre of water has to be pumped into one of these tubes, already quite full at atmospheric pressure, in order to reach 10 tons per sq. inch.

Passing now to the catalyst material itself, it is not at all difficult to find a large number of substances which will give a good activity for a short time if pure gases are used, but it is quite another matter to find a catalyst which will have a long active life under works conditions and with gases which are only technically pure. In this M. Claude has succeeded admirably, and he uses a material which is made from very cheap substances by a simple process. The basis of this catalyst material is peroxidised iron prepared by burning iron in oxygen, with the addition of other equally cheap materials which are added in order to increase its activity and its immunity from poisons. The oxide of iron is, of course, reduced by hydrogen in the catalyst tube. Other catalysts may perhaps be more effective scientifically than Claude's, but their use necessitates the most elaborate pre-

cautions in removing the last traces of impurity from the gases. Now one of the most deadly catalyst poisons which is liable to be present in hydrogen obtained from coke-oven gas or water-gas is carbon monoxide. Claude often uses hydrogen containing 2 or 3 per cent. of carbon monoxide, and this is made possible by an extremely simple device. A tube, similar to the catalyst tubes, is traversed by the gases on their way from the super-compressor to the catalyst tubes proper. This "protector" tube, as it is called, is filled with spent catalyst material which has become insufficiently active for further use in the catalyst tubes, and is heated to about 400° C. Any carbon monoxide present in the gases is converted into methane, $CO+3H_2 \rightarrow CH_4+H_2O$. Any oxygen present is at the same time burned to water. Obviously, in industrial working the percentage of carbon monoxide must be kept as low as possible, or a serious loss of hydrogen would occur; but it is quite easy to do this.

The robust nature and fool-proof quality of Claude's catalyst material are features which greatly impress visitors to his plant, particularly those who have already seen the Haber plants and who therefore realise the elaborate precautions which have to be taken, and the unceasing scientific control which is required to keep the Haber catalyst in working order.

The next question is how to remove from the system the ammonia formed in the catalyst tubes. Here again the use of superpressures results in striking advantages. On cooling the gases containing ammonia to ordinary temperature, a certain proportion of the ammonia liquefies and may be separated by gravity from the gas, this proportion depending upon the ratio of the vapour pressure of ammonia to the total pressure of the gases. Under the Haber conditions with 200 atmospheres the Germans find it necessary to inject water into the system and thereby recover the ammonia as an aqueous solution. This all consumes a good deal of power and involves no little complication of plant, and if the ammonia is required in the anhydrous state the liquor has to be distilled.

At 900 atmospheres, however, simple cooling by a coil immersed in water suffices to liquefy the whole of the ammonia save 2 or 3 per cent., and this small quantity is easily removed from the residual gases either by further cooling produced by the vaporisation of part of the ammonia, or by absorption in sulphuric acid.

The uncombined or residual gases from the last catalyst tube, after removal of this ammonia, are returned to the system.

Figure 5 shows the general arrangement of the plant. A and B are the hydrogen and nitrogen gasholders respectively, with meters, C, on the delivery pipes. The gases are mixed at the valve, D, and pass to the compressor, E, where they are compressed to 100 atmospheres. After going through an oil and water separator (not shown) the mixed gas is compressed to 900 atmospheres in the "super-compressor," F. Another oil and water separator, G, follows, and then the gas goes, *viâ* a heat interchanger, H_1, to the protector tube, J. Any water formed from traces of carbon monoxide or oxygen present in the gas is condensed in the cooler, M_1, and separated in the small separator, K. The pure gases next pass through the heat interchangers, H_2 and H_3, and the first two catalyst tubes, L_1 and L_2, in parallel, returning through the heat interchangers and the cooler, M_2, to the separator, N_1, where the liquid ammonia formed is withdrawn. The uncombined gas then continues through heat interchanger H_4 to the third catalyst tube, L_3, back through cooler M_3 to separator N_2, and the gas still uncombined goes to the last catalyst tube, L_4, and back to separator N_3 as before. The ammonia from the separators is blown into the collecting bottle, O, and from there to the storage cylinder, P.

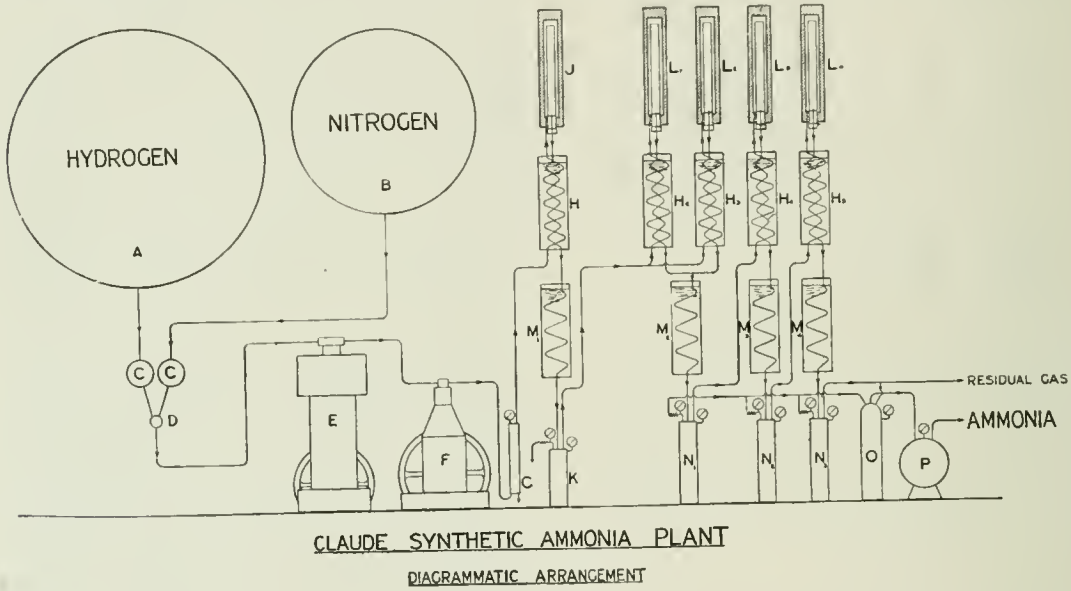


Fig. 5.

This completes the description of the Claude plant and process for making synthetic ammonia, and demonstrates it to be a remarkably simple, straightforward, and fool-proof way of attaining the desired result, and in fact a most striking contrast to the complicated and intricate Haber method.

The raw materials (hydrogen and nitrogen), and the power requirements of the process may next be considered. Nitrogen can be made in standard liquid-air machines at a cost of a few pence per thousand cubic feet, and since there is only one volume of nitrogen to three volumes of hydrogen,

the cost of it is not an important item. There are other methods of preparing pure nitrogen which will be dealt with later.

Hydrogen, however, is a big item in the cost, and cheap hydrogen is essential to the commercial success of synthetic ammonia. In this country, and at the present time, probably the most economical source of hydrogen is existing coko ovens. I say "existing" advisedly, because it would be another story if coke ovens had to be built at to-day's prices and a market found for the resulting coke.

The process adopted for separating the hydrogen from the coke-oven gas is a simple one of liquefaction in a modified form of liquid-air plant. The principle is fractional liquefaction, and not fractional distillation as in the liquid-air process. The coke-oven gas, after stripping in an ordinary by-product plant, is compressed to 25 atmospheres and passed through a tower containing water which dissolves most of the carbon dioxide, the last traces being removed by lime-water injected at the top of the tower. Were the carbon dioxide not removed, it would solidify and choke up the liquefaction apparatus. The compressed gas is then progressively cooled, the ethylene and similar hydrocarbons first liquefying, then the methane, after that the carbon monoxide, and finally the nitrogen, leaving only the hydrogen in the gaseous state. The nitrogen is liquefied at the top of the apparatus, and running down washes away the last traces of carbon monoxide which dissolve in it. The issuing hydrogen is allowed to expand while doing external work in an engine, and it is thereby cooled to about −210° to −215° C., and subsequently absorbs heat from the incoming coke-oven gas in a heat interchanger. The question of lubricating the cylinders of the expansion engine on this plant provided a nice problem. Petroleum ether makes a good lubricating oil for low temperature work, freezing only at −90° C., but this task was beyond it. Claude found a simple solution of the difficulty by mixing with the hydrogen a little nitrogen (the presence of which is of course quite harmless), whereby some of the nitrogen liquefies in the cylinders and forms an excellent lubricant. The manufacturing cost of hydrogen separated from coke-oven gas in this way is about 1s. 6d. per 1000 cub. ft. The remaining gases can be burnt in gas engines to produce power, and if the air admitted is reduced to a minimum the exhaust gases after removal of water and carbon dioxide are nearly pure nitrogen, thus providing another source for this material.

In countries where cheap water power is available, electrolytic hydrogen becomes very attractive.

A little consideration may now be given to the uses of synthetic ammonia, thus leading up to the question of the industrial future of the Claude process. The two main outlets for synthetic ammonia are, of course, nitrogenous fertilisers and nitric acid for making explosives. Taking the latter first, the national advantages of having large synthetic ammonia plants available in case of war are obvious, and it is ancient history to-day that without the Haber plants the Germans could not have carried on the war beyond a year, while we and our Allies were entirely dependent upon our command of the sea for supplies of Chile nitrate from which to make our explosives. The late Lord Moulton spoke most feelingly on this subject in the last public speech he made, one before a meeting of the Association of British Chemical Manufacturers on February 16 last, in which he alluded to the terror he felt during the four years of the war owing to the risk of German submarines concentrating on the nitrate ships. For peace time uses synthetic ammonia provides the cheapest sources of nitric acid, and solves the transport difficulty, for ammonia is comparatively easy to transport; it contains over 82% nitrogen against about 15½% in commercial Chile nitrate, and it can readily be oxidised to nitric acid at the place where the acid is to be used.

As regards fertilisers, the ammonia has to be "fixed" in some way, either with sulphuric acid to form sulphate, or by a modified form of ammonia-soda process to give chloride, an important by-product of this process being bicarbonate of soda. There are, of course, other possibilities for the future such as synthetic urea, and for special markets which must have nitrate of soda, this product can be made synthetically at a cost well below that of the natural product.

The following table shows the comparative power requirements and cost of power per ton of ammonia as between hydrogen obtained from coke-oven gas with steam power at ¼d. per unit, and electrolytic hydrogen with hydro-electric power at 1/25d. per unit:—

	Hydrogen from coke-oven gas. Steam power at ¼d. per kw.-hr.		Electrolytic hydrogen. Hydro-electric power at 1·25d. per kw.-hr.	
	kw.-hr.	%	kw.-hr.	%
Nitrogen	279	8·53	279	1·79
Hydrogen ..	1,287	39·35	13,590	87·27
Compression ..	1,530	46·78	1,530	9·82
Miscellaneous ..	175	5·34	175	1·12
Total ..	3,271	100·00	15,574	100·00
	Cost £6 16s. 3d.		Cost £2 11s. 11d.	

In order to obtain the advantage of the cheapest available source of power, the Cumberland Coal, Power and Chemical Co. has acquired a controlling interest in the Tysse water-power undertaking in Norway. This great power-station contains plant for producing 145,000 electrical horse-power, current being generated at 12,000 volts, 3 phase, 25 cycles. Including the actual running expenses of the power-station, upkeep, and depreciation on the purchase price, one horse-power will cost at Tysse £1, or less than 1/25 of a penny per kilowatt-hour. By securing the control of this undertaking, the company also acquires the extensive factories situated at Odda, about 4 miles from Tysse. These works, besides containing large manufacturing buildings, are well-eqnipped with workshops, foundries, stores, laboratories, and shipping facilities. At Odda, 100,000 electrical horse-power, transmitted from Tysse, is immediately available.

It is intended in the near future to erect a Claude plant to produce 156,000 metric tons of ammonium sulphate annually. Another works, with an initial output of 50,000 t. per annum of ammonium sulphate, is to be built near Workington, in Cumberland, adjacent to an existing coke-oven plant, the production being intended for home consumption.

Discussion.

The Chairman, Mr. J. A. Reavell (Chairman of the Chemical Engineering Group), said he wished to draw attention to the fact that this was the second meeting which the Chemical Engineering Group of the Society of Chemical Industry had had with the Institution of Mechanical Engineers, and they were fortunate in having with them that evening the President of the Institution, Capt. Sankey. He would therefore call upon Capt. Sankey to open the discussion.

Captain Sankey said he looked upon it as an honour to be asked to open the discussion at this joint meeting. He knew very little indeed, however, about this subject. His knowledge of chemistry

and his knowledge of mechanical engineering just enabled him to realise the beauty and simplicity of the chemical work and also the great ingenuity of the mechanical arrangements that had been adopted to meet the requirements of the Claude process. When he first heard of pressures of 900 atmospheres he had thought it must mean the most complicated mechanical arrangements, but to his astonishment, when he read the abstract of the paper, he found they were very far from that, because the mechanical part was so extremely simple, and, to his mind, that was one of the great attractions of the process. He would like to ask a question about the testing of the cylinders. It had been pointed out that Hooke's law was not obeyed after a certain pressure, but he would like to know if, after they had tested one of these cylinders and obtained the law of expansion they then released the pressure and then put it on again, would the same effect occur or would Hooke's law in that case be more nearly observed. He did not know whether the author had tried the experiment. In conclusion, Capt. Sankey said he would like to take the opportunity of observing how extremely important it was for mechanical engineers and chemists to hold joint meetings in this way. The Admirable Crichtons who were sufficiently good chemists and sufficiently good engineers to devise the apparatus required for the chemical industry must be very few and far between. Therefore, it was of extreme importance that these joint meetings should be held, and he hoped they would have a further and greater connection in some sort of direction or another in future. He could not define that at the moment any more closely. They must all feel that they had heard a description of a plant which was epoch-making and of which they would hear a great deal more in the future.

Dr. J. A. Harker, F.R.S., said he had been much interested in watching the progress of the Claude process right from its earliest beginnings to the present day. The author of the paper had emphasised some of the important differences between this process—which was described in detail for the first time—and the original Haber process of working, as operated by the Badische Co. While mentioning the Haber process, however, he ought to say, after some special opportunities for acquainting himself with the facts, that while it was quite true that the basic chemistry of the synthetic ammonia process was developed under Haber's general direction in the laboratories of the Technische Hochschule at Karlsruhe, yet it was in danger of being overlooked that this work was not, as was so often supposed, entirely a German achievement. He felt it only his duty to state that in his view its success was in very great measure due to the brilliant researches of two Englishmen, pupils and co-workers of Haber, viz., Dr. Robert le Rossignol, who was a Jersey man, and the late Dr. H. C. Greenwood. Mr. West had told them that one of the most striking differences between the Claude plant for a given ammonia output and that used in the Haber method, was that the Claude plant was very much smaller. He had stated that the space-time yield or the amount of ammonia produced in unit time by a given amount of catalyst by the Claude process was about 16 times as great as in the Haber method —1 litre of catalyst giving a yield of 5000 to 6000 grammes per hour instead of 350 to 400 grammes. That was certainly an achievement, and in the newer French method this result was brought about mainly by the adoption of the higher working pressure of 900 atmospheres instead of 200. He would like, however, to point out that increase of working pressure was not the only method by which greatly increased yields can be obtained. Some of those present would be aware that a long series of experiments on the problem of ammonia synthesis was carried out during the war period by the Nitrogen Products Committee under the auspices of the Munitions Inventions Department of the Ministry of Munitions. By the kindness of the Provost and Senate of University College, and especially by the enthusiastic help of Prof. Donnan, the Committee's Research Laboratory was located in the new Ramsay buildings of the college, and one of the first processes studied was that of synthetic ammonia production. By modifications of the original Haber method on lines completely different from those of Claude, the same kind of results and yields, as they had heard of from Mr. West, were now being obtained on a manufacturing scale. He himself said good luck to both systems. As soon as the world settled down to normal work and production, it was his firm conviction, after some considerable study of the economic situation, that the world's demand for increased supplies of cheap fertilisers would furnish an ample market for all that could be produced by synthetic and non-synthetic methods. There was one technical point to which he would like to refer. It was said that the actual proportion of combination in the Claude process was about 42% as against 13% in the Haber process. Was the 42% a working value or an equilibrium percentage? If it were an equilibrium value then the working value was bound to be lower. Another question was with regard to the series working. Was there evidence of any difference in the percentage of the gases combined in the first, second and third stages. Mr. West had said that there was rather over 80 per cent. combination over-all, and that by his formula he could calculate back to get the value of "a," but had M. Claude made any separate determinations to see whether the yield might be greater in the first, second or third vessel. It was obvious that the first catalyst had to stand the rougher treatment and would have the shorter life than the others, but supposing they had a good and new catalyst in all the vessels working normally, he would like to know whether Mr. West had anything to say as to the differences in the three.

Mr. J. Reavell (Ipswich), as a member of the Institution of Mechanical Engineers, supported what Captain Sankey had said about the advantage of holding these joint meetings. One important matter which appealed to all engineers was the lubrication of the compression apparatus. He must confess that he was surprised to hear that petroleum ether was satisfactory for lubricating purposes.

Captain Livens asked for information as to the composition and life of the catalyst.

Captain A. Egerton said there were two ways in which a process might be made a success. One was to add detail upon detail and make it complicated, although a success, and the other was by taking off everything that was irrelevant and leaving a simple and fine process. That was what M. Claude had done in his process. He had watched it since 1917, and every time he had seen it, it was more and more simple. On the other hand, in the case of the Haber process, exactly the opposite seemed to be the case, and little things had been added until it was a vast complicated process, although successful. It was not difficult, however, to make one's choice in favour of the simple process. It was the same in regard to the hydrogen process. In this case also M. Claude had removed everything that was complicated and made it somewhat on the lines of the Linde hydrogen process, but it was infinitely more simple than the Linde process. One of the slides had shown a comparison of the big Haber plant with the small Claude plant, but a better idea would have been given had the inside of the Haber plant been shown with all its complications. The comparison would then have been more noticeable.

Mr. W. H. Patchell, who also supported what had been said by Captain Sankey, as a member of the Institution of Mechanical Engineers, spoke with regard to the effect of hydrogen on steel pipes. He said there had been trouble with steam steel pipes and semi-steel pipes in some quarters, and it had been attributed to the effect of hydrogen. Could Mr. West say whether he had found anything of the sort in this plant, and whether hydrogen did act deleteriously on the steel at these high pressures.

Mr. West, replying to the discussion, said he had tried repeating the test on pressure, as mentioned by Captain Sankey, and found that if they did it a second time they got a straight stress-strain line up to where they had left off previously, and after that the curve began to go up again. Furthermore, if they left the pressure on for several minutes the stretch would go on slowly and gradually diminishing. In reply to Dr. Harker, he said the values were equilibrium values, but from the test results, of which Dr. Harker was well aware, M. Claude must get very near the actual theoretical equilibrium, and probably nearer than did Haber with his lower pressure. Haber had published figures, and he believed the actual combination which he got was 6 or 7 per cent. With regard to separate values in each of the catalyst tubes, he was not quite sure whether Dr. Harker meant the space time values or the actual work done in each tube. Of course, the third tube, as a matter of fact, had most of the work. The first two took a share between them, and taking 100 volumes of gas, there would be about 50 going through each tube, and taking in round figures a combination of 40 per cent., that gave 20 volumes combined in each first tube. Then they had got 60 volumes coming on to the next tube, and they had 40 per cent. of 60 in that, so that it was the third tube which had most of the work to do. After that, it was the first two, and the fourth tube had very little indeed. He did not think M. Claude had made any separate determinations of the space time yield for the particular tubes. As to petroleum ether, he did not think M. Claude had done any viscosity determinations. All he wanted was something which would stop his piston seizing up, but it might be that there was a liquid film which gave the effect in this case. Answering Captain Livens with regard to the catalyst, he was afraid he could not give any more information as to the composition than was in the paper. As regards its life, the life was so long that the cost of the catalyst material was infinitesimal. With regard to the question raised by Mr. Patchell, and the effect of hydrogen on steel, they always used pipes of the special nickel chrome steel when the gases were hot and they had been working without any trouble whatever.

Professor J. W. Hinchley, proposing a vote of thanks to the author, said the paper was a provocative one to some people, but unfortunately it had not succeeded in provoking them to come forward and say how *they* did it. He had hoped that Dr. Harker was going to say something of the way he had worked at University College, but he had not. However, the details of the Claude process had now been placed before them, and he hoped it would provoke other people to be equally open and to place all their cards on the table, because he did not think anything was lost by so doing. This paper was an example to our manufacturers not to be secretive about their work because he imagined they would get far more benefit by being open than by being secretive. He was not suggesting that Dr. Harker was secretive, because he had not the slightest doubt that the process was not his property. He was speaking of the people who were working other processes devoted to the fixation of atmospheric nitrogen.

The vote of thanks was accorded with acclamation.

Written Communications.

Captain C. J. Goodwin thought it was hardly possible in this discussion to adhere closely to the subject of the paper, as Mr. West obviously knew more about it than most of those present. He was somewhat surprised at the very low figure of £1 per horse power year given as the cost of electric energy at the hydro-electric works in Norway, and that was undoubtedly a determining factor in the economic future of a process which depended essentially on a supply of pure and cheap hydrogen. Even in America, and particularly at some of the new hydro-electric developments which he had recently visited, the cost was and would be many times as great, but there was a limit to the amount of plant which could be installed in Norway, and the question of costs in other countries was therefore of considerable importance in fixing the futuro price of synthetic ammonia in the markets of the world, and he hoped the author would amplify the information given in that direction. With regard to the figure of 1s. 6d. per 1000 cubic feet given as the cost of manufacturing hydrogen separated from coke oven gas, he would ask the author to supply accurate details as to how this was arrived at, as it seemed unduly low. The figure, no doubt, made suitable allowances for the revenue obtained from the various by-products, but unless this was stated together with the volume of the gases handled, the prospects of using coke oven gas could not be properly appreciated, as the by-product market was liable to considerable fluctuation. In the slides accompanying the paper, the author had shown M. Claude's plant making hydrogen from water gas and not from coke oven gases, and if it was a question of using water gas, it seemed doubtful whether so low a price could be obtained, as even with coke at 30s. per ton, water gas in England was to-day hardly obtainable at much less than 1s. per 1000 cubic feet. The very low cost of hydro-electric power in Norway would make it possible to raise steam at an economical figure, and in Canada a good deal of electric energy that would otherwise be wasted, was at present being utilised in that way. He had seen a three-phase electric boiler in Canada with a capacity of 15,000 kilowatts, and it was a remarkably small and cheap apparatus occupying less room than an ordinary Lancashire boiler.

The whole economic future of nitrogen fixation was a matter of national importance, and there could be no doubt that the Haber and Claude processes had almost completely upset all previous calculations and rendered the cyanamide and arc process relatively uneconomical. In the case of ammonia synthesis, we still, however, required three distinct processes, all requiring the highest technical skill, and while this was undoubtedly necessary for the production of ammonia, it seemed a pity to go through these many steps and the additional one of oxidation for the production of the nitric acid required by the explosives and other industries, and for the conversion of the ammonia itself into ammonium nitrate. Steinmetz had endeavoured to show that the arc process was not yet dead, while he personally inclined to the view that there was an even better future in store for an explosion process such as that of Häusser.

Just before the war he had inspected Häusser's experimental plant, but on account of the more immediate results obtainable from cyanamide and ammonia synthesis, the development of the process was postponed. The same conclusion was arrived at by the Nitrogen Products Committee in this country, although during the war the possibilities of the process were investigated theoretically by one of our most distinguished chemical engineers. Recently a plant of unit commercial size had been in economical operation in Germany, and had given

yields of about 100 grams of nitric acid per cubic metre of coke oven gas of 4000 Cal. and with an output of about 2 tons per day, and he ventured, therefore, to suggest that the Häusser process might prove an indispensable adjunct to processes like that of Claude. As oxygen was a by-product of the Claude process, and could be used to increase the yield of the explosion process, the two would fit together very well. He hoped the author would give some information as to the uses proposed for their by-product oxygen. A most important point in regard to the explosion process was the recent discovery of Bodenstein (Z. f. Elektrochemie, 1918, 183) that under a pressure of say 3 atmospheres the volume of the reaction and absorption towers required for the mixture of nitric acid and air could be reduced to 1½% of that required at atmospheric pressure. This was quite feasible with the explosion process, but not with the arc process. Special nickel chromium steels like "Vicronic," mentioned in Mr. West's paper, and those developed by Krupp under the name V2A, are capable of standing 50% boiling nitric acid, and would be suitable as they can be welded. Some 500 centrifugal pumps of V2A had been in operation., and he had seen some of them at work. In speaking of Vicronic as being a nickel chromium alloy, he presumed that a nickel chromium steel was meant, probably with a low carbon content.

Author's Reply to Written Communications.

In reply to Captain Goodwin, the figure of £1 per horse-power year given for the cost of power at Tysso is admittedly a very low figure, though absolutely correct in this instance. The reason for the lowness of the figure is that it was possible in this case to acquire very cheaply a hydro-electric undertaking built before the war. In any case of harnessing water-power to-day the cost would inevitably be much higher.

With regard to the figure of 1s. 6d. per 1000 cubic feet for hydrogen separated from coke oven gas, it should be pointed out that this refers to an existing coke oven plant which is an independent profit-earning concern. It is, therefore, only necessary to debit the hydrogen plant with the thermal value of the hydrogen removed from the gas, and at the time when this figure of 1s. 6d. was arrived at, this thermal value was considerably higher than it is to-day.

As to the point raised of Vicronic being a steel, this metal is considered by Messrs. Vickers to be technically not a steel.

DE-GASSING AND PURIFICATION OF BOILER FEED WATER.

By M. PAUL KESTNER.

Président de la Société de Chimie Industrielle de France.

A joint meeting of The Institution of Mechanical Engineers with The Society of Chemical Industry was held at The Institution, London, on Friday, March 4, 1921, at 6 o'clock p.m.; Captain H. Riall Sankey, C.B., C.B.E., R.E. (ret.) President, was in the chair, supported by Sir William Pope, K.B.E., F.R.S., President of The Society of Chemical Industry.

The PRESIDENT remarked on the nature of the meeting, because it was a joint meeting between the Institution and the Society of Chemical Industry. He was glad to announce that Sir William Pope, the President of that Society, and several important members of the Society were present. M. Paul Kestner, the author of the paper, was the President and Founder of the Society of Chemical Industry in France. He was a very well-known French chemist and an engineer, who had done much work in connection with sugar refining, multiple evaporators, and other matters of a similar nature. He would ask M. Kestner to present his paper.

How to feed boilers with water and to enable them to run for long periods under steam without scale or corrosion is the problem which becomes more urgent every day, and further, it is a problem on which a great economy of fuel and material depends. It is certain that a large number of technical men have already attacked the problem —frequently with considerable success, but never with complete success. The old lime and soda purifying apparatus, of very large dimensions, has been gradually replaced by apparatus of a much smaller size in which, thanks to the arrangements which will be discussed, the rate at which purification is effected has been increased. Lately, purification has been done by heating, which has accelerated the process. In spite of these efforts, some kinds of water have proved refractory to treatment; after addition of lime and soda, when raised to high temperatures, they have still given rise to scale in the boilers. A process which has been effective with one kind of water has given only indifferent results when used with another water. Another process which has eliminated all calcareous salts from the water has, in some boilers, produced a concentrated solution of soda. Perfect purification is rarely obtained.

There is, however, a process which ensures the prevention of the formation of mud and incrustation in boilers. This process, which is that of continuous blowing-down, will be considered in this paper; it is necessarily used in conjunction with the hot purification process, and the combination constitutes a complete method of treating the boiler feed-water.

Purification at high temperature. It is unnecessary to recall the advantages of feeding boilers with hot water. In addition to those advantages due to the thermal side of the question, there are those produced by the reduced solubility of gases in water at high temperatures, as will be shown. Purification at ordinary temperatures, whether by the use of soda alone or lime and soda, requires considerable time, owing to the slowness of the reaction and the fineness of the precipitate formed, which settles very slowly. The reduction in the time required for the same reactions in hot and cold liquid is shown in the curves, Fig. 1, which has been taken from a catalogue published by Messrs. Cochran and Co.

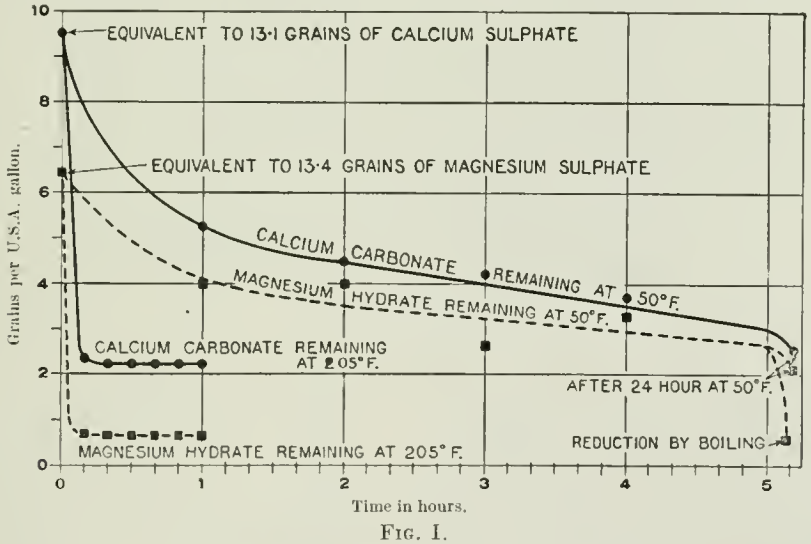


FIG. I.

Curves showing reduction in time required for the same reactions in hot and cold liquids.

Ingenious processes have been invented for enabling the speed of these reactions to be increased and to hasten precipitation in cold water. This consists in bubbling through the sediment in the water to be purified after the addition of the reagents. Under the influence of the large quantity

of old residues, the size of the particles that are formed increases; the small particles that come into existence are attracted by the larger ones that have already formed which absorb them, and this precipitate, which consists of much coarser material, settles very much more rapidly. Owing to the increased speed of settling, the reagent quickly becomes poor in one of its constituents, and the equilibrium of the reaction is favourably disturbed. This process allows the rate of purification to be increased, and hence the dimensions of the apparatus can be reduced; but, on the other hand, a difficulty is generally introduced where agitation of the sediment is obtained by a stream of water which carries air with it. This causes a considerable increase in the amount of oxygen contained in the water that is to be purified; under the conditions met with in practice saturation occurs almost instantaneously and gives rise to corrosion in boilers.

In any process for softening water, a rise of temperature gives the following advantages:—

(1) *Increase of the speed of reaction.* For the same initial concentration, equilibrium is obtained, as is well known, more rapidly at high temperatures than at the temperature of the air. This follows from the laws of Guldberg and Waage, Gibbs and Le Chatelier, to which there is no reason to refer further. We need only mention that at the temperature of absolute zero, *i.e.*, – 273° C. reactions have no speed at all, and that any rise of temperature increases the speed of obtaining equilibrium in any reaction. In the case of the reactions with which we are concerned, and which are of the character of double decomposition, equilibrium is only slightly affected by rise of temperature, but the speed at which it is obtained is enormously increased.

(2) *Diminution of the viscosity of the water.* The coefficients of viscosity decrease very rapidly, as is shown by the following curve, Fig. 2.

Viscosity is the chief resistance which is opposed on the one hand to molecular attraction of the particles of the precipitate towards each other in the formation of larger aggregations, and on the other hand to the settlement of the particles formed from suspension in the solution in which the precipitate has been produced till they reach the bottom of the containing vessel. The coefficient of viscosity is the tangential force required to displace an unit area at unit velocity in the medium under consideration. It will be seen, therefore, that the rate of forming a granular precipitate depending on molecular attraction will, other things being equal, be directly proportional to the coefficient of viscosity. The velocity of falling of a spherical particle having a density greater than water will be given by Stokes's formula.

$$V = \frac{2}{9} \cdot \frac{D-d}{N} \cdot g \cdot r^2$$

in which V is the velocity, D the density of the particle, d the density of the water, g the acceleration due to gravity, r the radius of the particle, and N the coefficient of absolute viscosity.

If we examine the curve of coefficients of viscosity, we shall find that this maximum velocity of falling will, under some conditions, be four times as great at 100° C. as it is at 12·5° C. We may therefore deduce that the deposition of a sediment will be four times more rapid with water at boiling point, and that the volume of the purifier may be reduced in the same proportion.

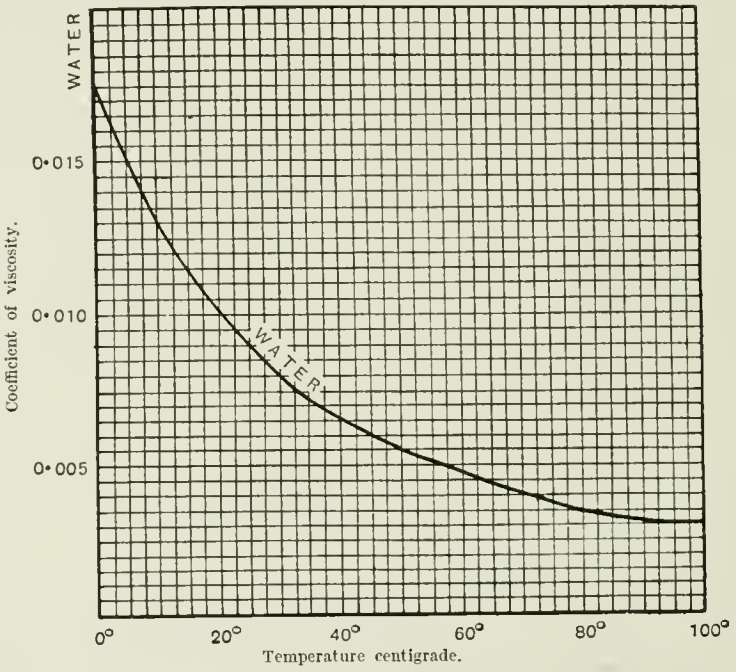


FIG. 2.

Curves showing rapid decrease of the coefficients of viscosity.

(3) *Decomposition of the Bicarbonates of the Alkaline Earths.*—The direct effect of temperature on bicarbonates of the alkaline earths is to decompose them at 70° C. into carbonic acid gas and the carbonates of the alkaline earths, without the addition of any reagent.

(4) *The Diminution in the Solubility of Sulphate of Calcium.*—Sulphate of calcium becomes reduced at higher temperatures, and the precipitation of part of this salt is effected without the intervention of chemical reagents. This reduction is of very slight importance in the process using continuous blowdown, just as the increase of solubility of calcium carbonate is a negligible factor. We shall see later

why these facts have so small an influence in the particular case under consideration.

Continuous Blow-down.—When a boiler, the feed-water for which is purified by any method, has been running for some time, all the salts carried in with the feed-water are concentrated in it. These salts are only alkaline salts when the water has been thoroughly purified, and the calcareous sediment of the water has been incompletely purified. In general, a definite amount of calcium carbonate always remains in solution in the water. When the concentration produced by evaporation becomes so high that, notwithstanding the rise of temperature, this carbonate remains insoluble, it is then deposited on the walls of the boiler and in the tubes, forming incrustation, which is the harder the more slowly the deposit is made. In boilers generating steam very rapidly, these deposits are formed more rapidly than in slow evaporation boilers, and notwithstanding the violent circulation of the water, sediment may accumulate in the tubes. These sediments give rise to overheating and to very hard incrustation.

To avoid these difficulties, in actual practice, the boiler is blown off, that is, at the lowest point on the boiler shell, the water containing the collected mud is drawn off and turned into the drain. This water is at the pressure and temperature of the boiler, and, in blowing off, a large quantity of heat is lost. To be effective, blowing off must take place to the extent of a large actual quantity of water, though the amount is small in proportion to the total contents of the boiler. Continuous blow-down allows these calories to be saved. Instead of sending the blow-off direct into the drain a relatively small quantity is caused to leave the boiler continuously from its lowest point. This water cools in giving up its calories to the feed-water in a feed-water heater of ordinary pattern; the mud is deposited in a trap; it loses in cooling a certain proportion of the salts which had been retained in solution owing to the high temperature of the liquid. Then, the salts which would cause incrustation, mud and deposit having been collected and removed, the liquid is mixed with the feed-water and returns into the boiler. It will thus be seen that there is no loss of calories due to the blow-off, and that neither deposition nor scale can be caused in the boiler. There is, however, another cleansing operation to perform. It is the removal of the mud deposit at the blow-off outside the boiler. This removal is only of small magnitude, because the apparatus in which precipitation takes place has a conical bottom suited to this purpose; and, further, the water in it is not under pressure; it removes only a few calories because the blow-off water is at this moment already considerably cooled. This small amount of extraction at low temperature completely suffices to limit the concentration of the soluble salts before danger from the raising of the boiling point of the water commences.

If the boiler has been blown down to the extent of 20% of its contents in an hour, this means that its contents are renewed every five hours. This is impossible to effect by any other process, and it ensures the elimination of all mud that has been produced before it has had time to accumulate. This shows the great advantage presented by the process of continuous blowing-down in itself, this advantage being twofold when the feed-water is purified by means of soda; in fact, in this case, the calcareous and magnesium bicarbonates contained in the water give, by double decomposition with sodium carbonate, sodium bicarbonate and calcareous carbonates according to the reaction:—

$$(CO_3)_2CaH_2+CO_3Na_2=2CO_3NaH+CO_3Ca.$$

The bicarbonate, which is unstable at the temperature at which the purification in hot water takes place, decomposes, at any rate partially, giving a neutral carbonate and carbonic acid gas, according to the equation

$$2CO_3NaH=CO_2+H_2O+CO_3Na_2.$$

The water which enters the boiler contains, therefore, carbonate and bicarbonate of soda. These salts are in quantities equivalent to the calcareous salts that have been eliminated. In the boiler the bicarbonate is decomposed, giving a carbonate, as has been seen above, and then the carbonate in its turn is dissociated in the presence of water, giving caustic soda. There is thus another reversible condition of equilibrium in functions of the initial concentration on the one hand and of the temperature on the other. This may be represented in the following form:—

$$CO_3Na_2+H_2O=2NaOH+CO_2.$$

Thus hydrolysis gives more caustic soda, the higher the temperature.

With continuous blowing-down, water containing caustic soda and carbonate of soda is therefore drawn from the boiler. If this solution be mixed with untreated water, the caustic soda and the carbonate they contain will purify the water; the carbonate in the same manner as previously mentioned, and the caustic soda in the following manner:—

$$(CO_3)_2CaH_2+2NaOH=CO_3Na_2+2H_2O+CO_3Ca.$$

The caustic soda reacts on the calcium bicarbonate as would calcium oxide, but much more rapidly. New products will then be formed, namely, sodium carbonate and bicarbonate, which will be returned to the boiler.

If the quantity of blowing-down is calculated in such a manner that it brings down the quantity of soda necessary in a given time for the purification of the water entering the boiler in the same time, it will be seen that it will not be necessary to add any carbonate of soda for purification of the temporary hardness of the water. A closed circuit will be formed containing a certain quantity of soda, which will never be exceeded. The soda is never concentrated in the boiler, because no fresh soda is introduced. The concentration in the boiler depends on the proportion of blow-down in relation to the feed-water. The less the quantity of blow-down, the greater will the concentration become, so that the same quantity of salt will be contained in a smaller volume.

It is therefore of the greatest importance to combine purification by soda with the continuous blow-down. This allows of more economical use of the chemical reagents necessary for freeing the water from the bicarbonates of the alkaline earths. Consequently, owing to the circulation of the blow-down through the purifier, there is a blowing through the deposit favourable to drawing-off and purification at high temperature by using the calories that would be thrown away in the drain under ordinary methods of blowing-off. The concentration of the soda in the boiler is limited, and neither deposit nor incrustation can be produced. The consumption of soda is reduced to that necessary for the decomposition of non-carbonates. The author has devised the following apparatus, using a blow-down on the preceding principle, Fig. 3.

This apparatus consists of a steam separator, S, into which the blow-down enters in the form of a mixture of steam and water. The liquid from the blow-down falls into a central reservoir, P, in which it gives up its heat to the water surrounding it in the annular chamber, B, into which it is decanted.

The steam from the blow-down rises in the reheater, R, in which it reheats the feed-water, which loses a part of its bicarbonate, owing to the rise of temperature. The water thus heated falls through the tube, T, and mixes with the blow-down as it leaves the central chamber, and then rises in

the annular chamber, B, in which precipitation and settlement take place. Reheated by admixture with the blow-down, as well as by exchange of heat with the central vessel, the purified water passes through the filter and enters the boiler. The soda is distributed in the apparatus by means of a water-meter, J, which admits it in proportion to the new water (make-up feed). The valves, V and V_1, allow the mud to be separated. This apparatus enables the thermal and chemical properties of the blow-down

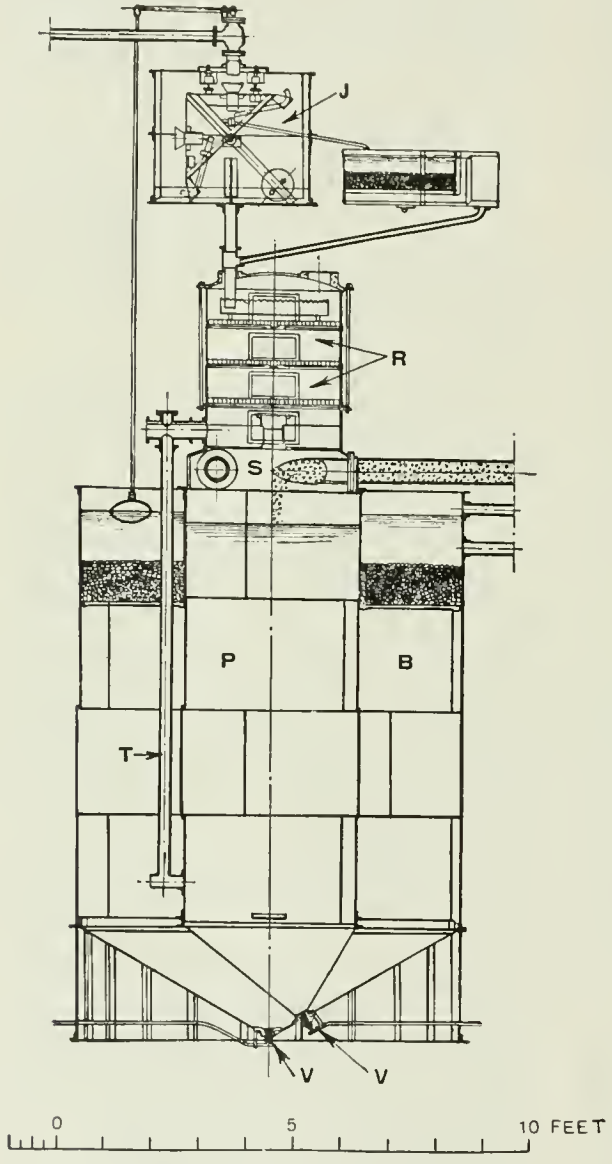


Fig. 3.

Blow-down apparatus enabling the thermal and chemical properties to be saved. (Kestner.)

to be utilised, or, in other words, heat and chemicals are saved.

Position of the Blow-down.—It is necessary to arrange the blow-down at the lowest possible level on the boiler without risk of emptying it. A simple arrangement is recommended by which, when the water of the boiler falls below a certain level, the siphon action of the blow-off is destroyed. At this moment steam leaves by the inlet and calls attention by sounding a whistle. In any case it is impossible to empty the boiler by the continuous blow-down, Fig. 4.

On the pipe of the continuous blow-down and the outlet from the boiler, a reducing valve is placed, which reduces the pressure of the blow-down from that of the boiler to a pressure of between one and two kilogrammes per square centimetre (15 to 30 lb. per sq. in.) before it enters the purifier. In order to allow of the amount of the blow-down being regular,

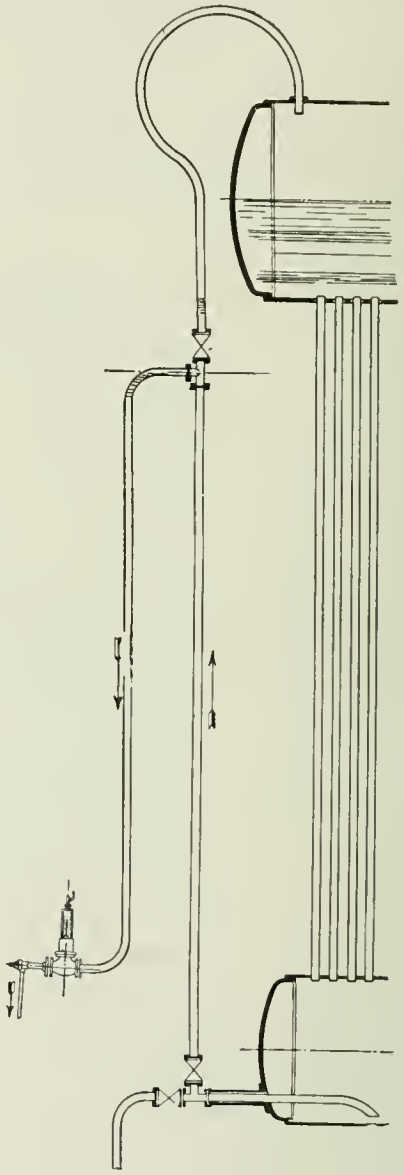


Fig. 4.

Continuous blow-down.

a meter is placed at the outlet of the boiler and is fitted with a regulating valve, Figs. 5 and 6.

Proportions of the Blow-down.—The proportions of the blow-down are calculated according to the temperature to be maintained in the purifier. It is always desirable that the temperature at which purification is effected should not be less than 70° C. According to the working pressure of the boiler,

various rates of blow-down may be used, and these can be obtained by the curves, Fig. 7.

The temperature at which the water leaves the purifier, although very nearly 100° C., allows the use of an economiser, as has been shown by recent researches in France.

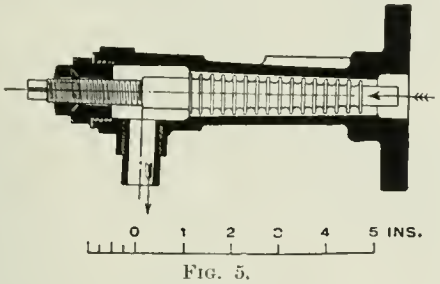


FIG. 5.

Regulating valve.

Central Stations Using Distilled Water.—It has just been shown how the system of continuous blow-down can be applied to a boiler using purified water. A case offering still greater interest is that of central stations using distilled water.

The distilled water of industrial appliances differs greatly from the distilled water of the laboratory. In these appliances, it is necessary to blow down the boilers as a concentration of the boiler contents arises, due to the entry of salts through accidental leakages of impure water in the condensing plant; thus the water that has been distilled contains numerous soluble salts, present in smaller proportions than in purified water, but by no means negligible. The following relationship is found to exist between the water in the boiler and the water of distillation:—

	Untreated water.	Distilled water.	Proportion of remaining salts.
Total solids at 110° C. in parts per 1,000,000	5,427	26·4	$\frac{1}{205}$
Total solids at 400° C. in parts per 1,000,000	4,414	8·4	$\frac{1}{525}$
Organic substances in parts per 1,000,000	1,013	18·4	$\frac{1}{55}$
Silico SiO_2 in parts per 1,000,000	243	1	$\frac{1}{243}$
Oxides of iron and alumina in parts per 1,000,000	200	3·2	$\frac{1}{62}$
Lime CaO in parts per 1,000,000	73	2	$\frac{1}{36}$
Magnesia MgO ,, ,, ,,	42·4	1	$\frac{1}{42}$
Chlorine ,, ,, ,,	2,583·8	Traces	0
Sulphates SO_3 ,, ,, ,,	407	0	0
Hardness ,, ,, ,,	24	0·54	$\frac{1}{44}$

In boilers using distilled water, this water in time becomes a fairly concentrated liquid. Organic matter—oils and grease particularly—is present in considerable quantity, and of these only a small quantity is necessary to produce priming. It is, therefore, necessary to avoid this concentration, and continuous blowing-down is consequently applicable in this case also.

Boilers working with distilled water therefore require to be blown down occasionally, but, owing to the small amount of salt that they contain, it is useless to remove a large quantity of liquid. These boilers are consequently blown-down to the extent of 1 or 2% only, which means that the quantity of saline solution is replaced in the boiler by distilled water. By this method the increase in the quantity of dissolved matter is necessarily limited. It could not, for example, exceed fifty times the concentration of the distilled water taken from the condenser, in the case of a boiler blown down to the extent of 2%. In the case quoted above, this corresponds to a dry residue of 1·3 grm. per litre, which is certainly not negligible. The water blown down is of no special interest so far as its chemical composition is concerned; but it is hot water which enters the distilling apparatus without loss of its calories.

Even when an evaporator is used, the continuous blowing-down method can be adopted. A certain amount of saline or impure water can be drawn from the boilers and passed into the evaporator; this latter returns it to the boiler after it has been freed of the greater portion of its salts. It will be seen later how the simultaneous use of continuous blowing-down, in conjunction with degassing, allows of the establishment of a complete closed water circuit in a central station using distilled water.

Experience has shown that boilers fed with distilled water as well as those fed with purified water are liable to deterioration due to corrosion of the plates. The interior of the boiler, which is smooth and clean when put to work, becomes covered in places with a thin coating of rust; the small patch of rust increases in size little by little; the rust forms a scale of somewhat spongy nature, and when the boiler is put off work, or frequently even while

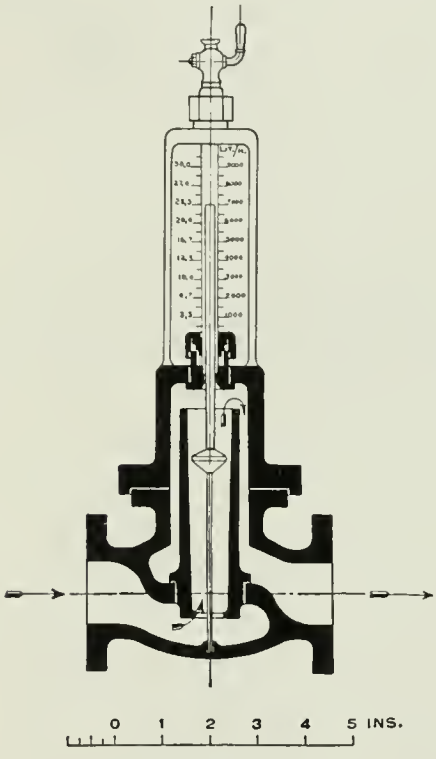


FIG. 6.

Flow indicator.

it is still at work, for various reasons this scale ceases to adhere to the plate and falls from it. In its place there is formed a slight depression, caused by the disappearance of the metal which has entered into combination in forming rust; the depression becomes again rusty, and eats further into the plate by repetition of the process. Some boilers, after having been a certain time at work, have their plates pitted all over so that they recall the battered and dented walls of a village that has been under fire during the war. The strength of the boiler is undermined, and it becomes necessary to renew the damaged plates, or even the whole boiler.

Although the commencement of corrosion does not always become visible, it is nevertheless the cause of serious damage to boilers and shortens their life. Owing to the high first cost of a boiler, an important economy is effected by extending its working life by one or more years. This can be done by preventing corrosion.

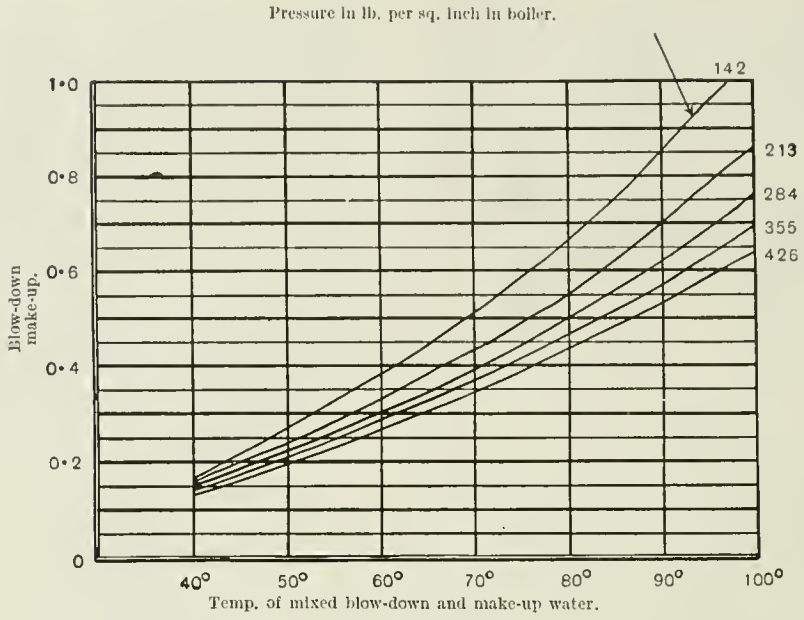


FIG. 7.

Curve showing temperature of water leaving heater settler. (*Water purification.*)

The Causes of Corrosion.—What is the origin of corrosion? In the case of boilers using distilled water there can be no doubt. Oxidation of the plate can only be produced by oxygen dissolved in the feed-water and set free in the boiler. When the rate of evaporation is steady and the boiler is in regular work, the dissolved air is released at those portions of the boiler in which the water has not reached the boiling point, or where the circulation is sluggish. At these places bubbles are formed which cling to the surface, and leave their oxygen in combination in the form of rust. There is

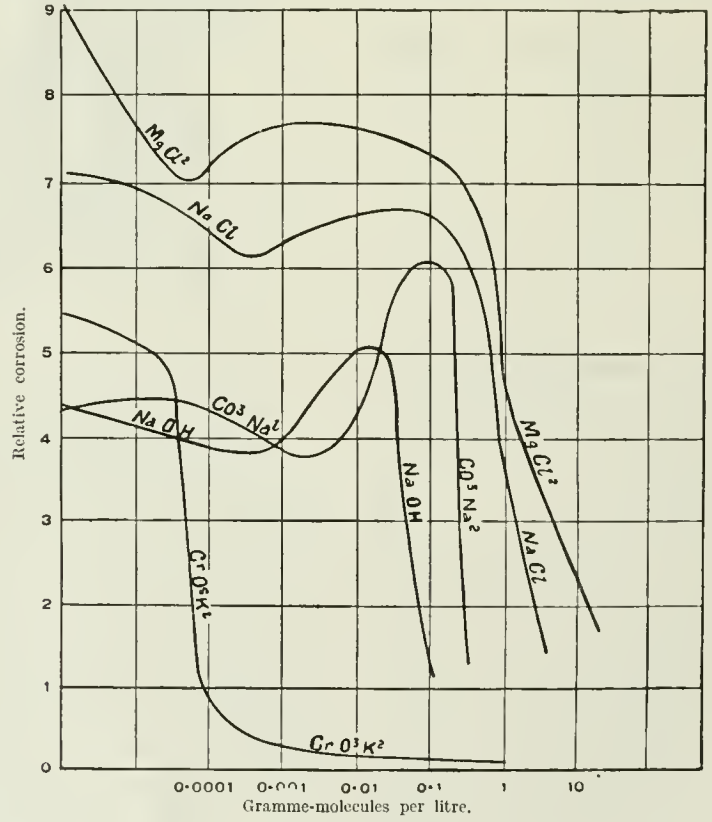


FIG. 8.

Relation between corrosive power and concentration for a number of solutions.

also the freeing of air-bubbles at the water level when the rate of evaporation is reduced. It is well known that intermittent working is the most destructive cause of all.

The combination that takes place between the oxygen and the iron can be explained in several ways, as may be seen by reference to the work of Newton Friend.* The usually accepted theory is that oxygen, in the presence of water vapour, combines directly with iron according to the following reaction:—

$$2Fe+3H_2O+3O=2Fe(OH)_3.$$

Treadwell's theory, according to which carbonic gas acts as catalyser, is also interesting, and leads to the same result:—

$$Fe+CO_2+H_2O=CO_3Fe+H_2$$
$$2CO_3Fe+3H_2O+O=2Fe(HO)_3+2CO_2$$

It is, however, to be noted that even when there is no carbonic acid gas, so far as our methods permit its presence to be detected, rusting occurs. This is disputed by some authorities, but whatever the actual process, it cannot be denied that oxygen is the cause of rust. Numerous electrical theories have been proposed to explain the oxidation of iron. These are not, however, applicable to the case of boilers using distilled water, where electrolytic action is minute and the weakness of the saline solution in the boilers is unfavourable to the production of local currents.

All these theories, taken individually, appear very plausible and might form the subject of a lengthy chemical investigation, into which it is not proposed to enter, as it is of academic interest only.

If the action of oxygen is obvious and well known in boilers using distilled water, it is not so clear in the case of boilers using purified water, or it may be considered less important on the ground that the concentration of salts, obtained under this system of working, forms the principal factor in the eating away of the plates. This erroneous view depends generally on an incomplete appreciation of the facts; it has, moreover, been proved that although highly concentrated solutions attack the gun-metal fittings more severely, their action on iron is greatly reduced.

If we examine the corrosive effect of saline solutions, we find that in general it begins by increasing in proportion to the strength of concentration up to a critical point at which it attains a maximum; the effect then decreases rapidly as the concentration proceeds. The diagram, Fig. 8, shows the relation between corrosive power and concentration for a number of solutions. From these curves it appears that there is a certain critical concentration which must be exceeded in working boilers. In practice, after a few hours' work, a boiler using purified water will have already attained a concentration fully one hundred times the critical amount. Concentrated solutions may, therefore, give rise to a higher boiling point, to priming, and to the deposition of crystals, but corrosion must not be included in the list.

Electrical action may have some influence on the preceding. In fact, stray currents may find suitable conductivity in the salt solutions, but boilers are not made of a single piece of plate, and the interruptions caused by the joints tend to localise the effects of the current. Moreover, it appears probable that if currents of sufficiently high voltage existed, they would soon cause serious damage, the only remedy for which would be the adoption of insulation of the boiler. Generally the boiler seatings are very dry and offer no facility for the passage of electric currents. It may happen that galvanic pairs are formed between parts of the boiler which differ in chemical composition or physical nature, but these pairs are generally of but feeble strength and their electrolytic effect would be slight and confined to small areas.

There is still the question of electrolytic dissociation. This occurs much more easily in a dilute than in a concentrated solution, which confirms the previous statement; in general it is of much less importance than the phenomena to which corrosion is attributed. Hydrolysis may have much more important effects. A particularly important case, and the best known, is that of magnesium chloride, which gives rise to the production of hydrochloric acid, and is liable to cause indefinite corrosion. Magnesium chloride in the presence of water may be decomposed into magnesia and hydrochloric acid, which attacks the plates vigorously.

$$MgCl_2+H_2O=MgO+2HCl.$$

Fortunately this salt only occurs occasionally, and is sufficiently well known for special care to be taken where the feed-water contains magnesium chloride. Acids attack the plates, but, apart from the case just mentioned, the only acid found in boilers is the carbonic acid introduced in the feed-water. Carbonic acid alone has only feeble action on iron; in the laboratory a cold concentrated solution of the gas produces only a few bubbles of hydrogen on a piece of iron immersed in the solution. If, however, oxygen is present, the metal is attacked much more rapidly, because the ferrous carbonate formed decomposes into rust and carbonic acid, which recommences the action. In this case also it is oxygen that produces the corrosion.

Oxygen comes into play still further when the water contains salts capable of combining with oxygen and forming unstable oxygen-compounds which can part with their oxygen to form other oxides. Among these salts some, particularly certain oxychlorides, are known and have been separated; but the hypothesis, that certain oxy-salts that have not yet been separated may be present, affords an explanation of the rapid eating away of iron by sodium chloride in the presence of air, whereas solutions of the same strength, absolutely free from oxygen, can be kept in contact with the metal without effecting any change whatever.

Thus it appears that it is the solution of atmospheric oxygen in water that is the chief cause of corrosion, whether the feed is distilled water or purified water. This is supported by the fact that corrosion is often more serious in boilers using distilled water, in which dissolved oxygen can be the only agent, than in boilers using purified water.

The remedy therefore appears to be very simple, namely, the elimination of the dissolved oxygen.

Oxygen in solution in the feed. Atmospheric air (and therefore the oxygen that it contains) dissolves in water almost instantaneously up to the point of saturation for the particular temperature and pressure. The solubility of air in water is proportional to the pressure, and inversely proportional to the temperature; it follows Henry's law, which, though approximate, is very nearly true for the case of air mixed with a gas that is not very soluble in water. Each of these gases dissolves in proportion to its share in the pressure, as though alone; thus air contains four volumes of nitrogen to one volume of oxygen; the share due to the nitrogen is 0·8 and that due to the oxygen is 0·2.

At ordinary temperatures and pressures 18 cm³ of pure nitrogen and 35 cm³ of pure oxygen form saturated solutions with 1 litre of water. The quantities of each dissolved from air are therefore:

Nitrogen: $0{\cdot}8\times18=14{\cdot}4$ cm³.
Oxygen: $0{\cdot}2\times35=7{\cdot}0$ cm³.

The air dissolved in water will therefore contain, roughly, 33 per cent. of oxygen, and be twice as rich in this gas as atmospheric air. Laboratory

* "The Corrosion of Steel and Iron."

tests have shown that the quantity of oxygen dissolved in water varies slightly with the nature of the water; distilled water absorbs more than fresh water, and soft water more than hard water. Distilled water absorbs 7·8 cm³ per litre at 20° C., 2·1 cm³ at 100° C. The water supply of Paris contains 6·06 cm³ per litre at 20° C., 4·3 at 55° C. and 2 cm³ at 100° C.

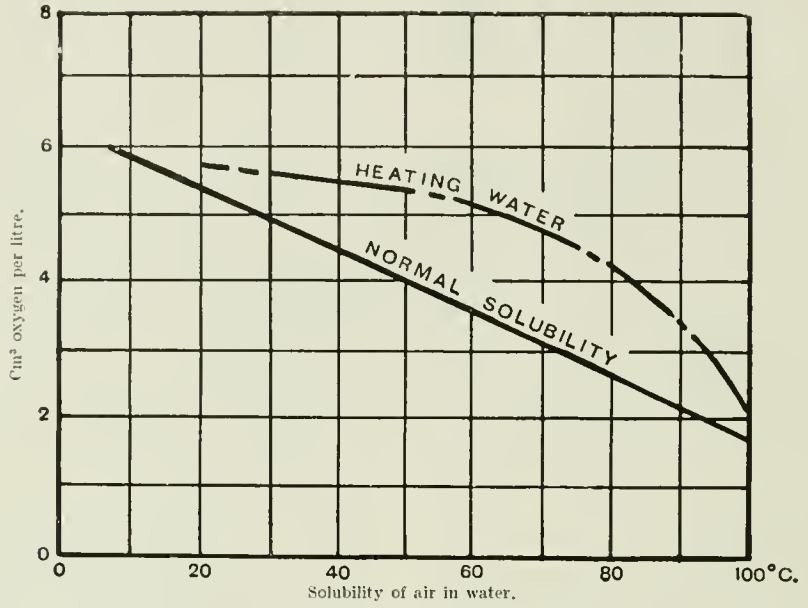


FIG. 9.

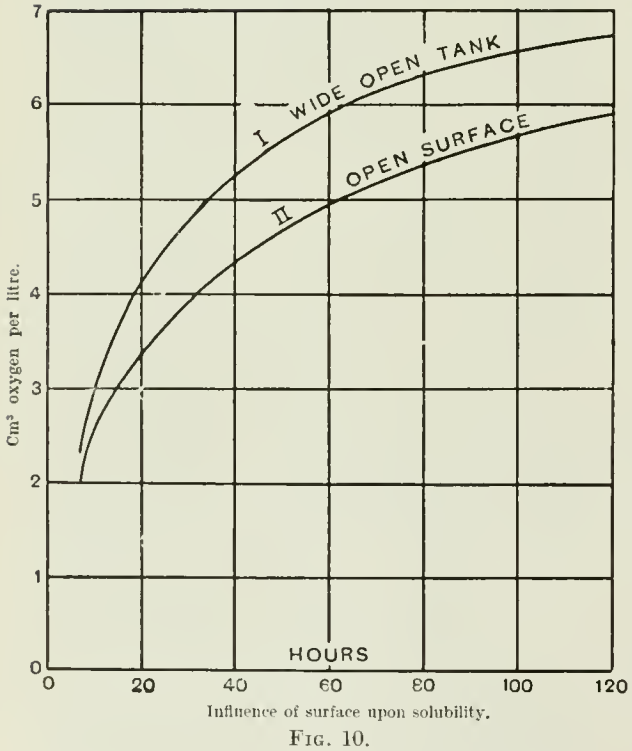


FIG. 10.

When water is heated, supersaturation occurs. Nothing is given off up to a temperature of 75° C. at which bubbles begin to form and rise until boiling takes place, when the bubbles are disengaged continuously. Air is only driven off continuously when the water is agitated incessantly from the commencement. When water that has been boiled is cooled, the inverse takes place; a litre of water absorbs, for instance, 2·3 cm³ at 44° C. and 3 cm³ at 20° C. If the water is agitated in air the water is agitated in air the false state of equilibrium is distributed, and the water becomes saturated up to 6 cm³ of oxygen, Fig. 9. The straight line is the normal solubility obtained when the water is continuously agitated, and the curved line gives the contents at different temperatures without agitation.

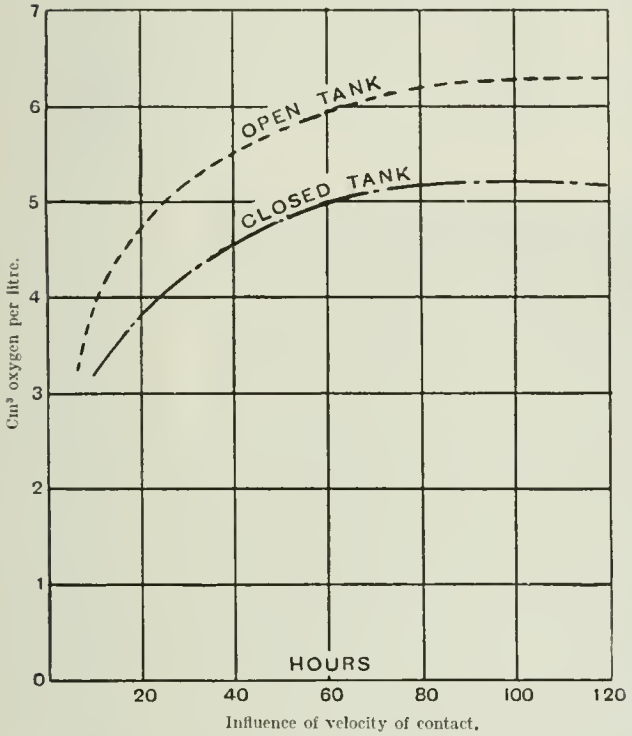
Thus conditions of unstable equilibrium may occur which cannot exist when the liquid is agitated, and in every case equilibrium is obtained for the particular temperature and pressure considered. If water is contained in a deep vessel, a sample taken at any moment from the surface contains the same percentage of gases as a sample taken from below; the diffusion of oxygen in water is extremely rapid. The area of the surface exposed to the air of water that has been boiled has an influence on the rate of absorption. Thus the water resulting from the condensation of the vapour in the air contains 4 cm³ of oxygen per litre at 60° C. The same quantity of water or condensation exposing only a small surface to the air contains only 3 cm³ of oxygen per litre at the same temperature. According to Hohn, the surface affects the rate of absorption of the oxygen, as is shown in curves I and II obtained with a surface II seven times that of the surface I, (Fig. 10).

The possibility of changing the air also has an influence. If the reservoir is closed, but not hermetically, the oxygen is absorbed less rapidly than it could be from the open air, in which case saturation would be almost instantaneous, owing to the contact and the movement of the water. Hohn gives the following curves, Fig. 11.

Method of obtaining the oxygen content. All the methods of analysis that have enabled the data given above to be obtained and the determination of complete absence of gas have been made by Max Müller's method in which certain modifications have been made.

General description. The water is collected in a vessel protected from the air. Manganese sulphate

is dissolved in it, and by the addition of sodium hydrate the hydrate of manganese monoxide is precipitated; this absorbs the oxygen contained in the water, transforming it into an oxy-salt and the dioxide. This is treated with hydrochloric acid, which reacts on the oxides of manganese, giving off a quantity of chlorine equivalent to the quantity of oxygen absorbed. The freed sodium chloride reacts on potassium iodide and sets free the equivalent of iodine. Titration by hyposulphite of soda gives the quantity of iodine and enables the equivalent amount of oxygen to be determined.



Influence of velocity of contact.

FIG. 11.

Method of operation. It is important, when it is a question of determining the quantity of oxygen in water containing but little, to avoid any contact of this water with the air. For this it is only necessary to put a small quantity of paraffin at the bottom of the 2-litre flask in which the water will be received and to dip the tube bringing the water below its surface. Thus, during the whole operation, an insulating covering will be provided over the surface of the water.

After the water has been collected in the flat-bottomed 2-litre flask, 1 gramme of sulphate of manganese is added and the neck is immediately closed with a rubber stopper fitted with a funnel having a stop-cock and a glass tube connected by a rubber tube to another glass tube of larger diameter, carried on a stand.

On the principle of connected vessels, it is possible, by raising or lowering the tube, to change the pressure in the flask and to cause it to suck in liquid contained in the funnel fitted with the stop-cock. It is necessary that there should be no air bubbles in the apparatus, and this is easily attained after a little practice. When the sulphate of manganese has been dissolved, 5 cm³ of sodium hydrate in solution is introduced through the funnel of the stop-cock and the flask is shaken. A dirty white precipitate is formed, turning more or less yellow, according to the quantity of oxygen contained in the water. At the end of a quarter of an hour the absorption of oxygen is complete.

The next operation is to introduce 2 cm³ of solution of 50 per cent. of iodide of potassium free from iodate through the funnel fitted with the stop-cock, and then to add 20 cm³ of concentrated hydrochloric acid. The flask is shaken until the precipitate has dissolved; the liquid takes a yellow colour, darker as the quantity of iodine freed is greater. The flask is then emptied into a bath and the iodine determined by titration.

The most practical method consists in preparing a standard solution of iodine containing 22·74 grammes per litre; that is, of such strength that a cubic centimetre corresponds to a cubic centimetre of oxygen. A solution of hyposulphite is prepared of such strength that it corresponds practically volume for volume with the solution of iodine. The number N of cubic centimetres of this solution corresponding to 20 cm³ of iodine is determined experimentally. To the solution of iodine contained in the bath N cm³ of hyposulphite is added, and then a small amount of starch, and the iodine is titrated again until a permanent blue colour is obtained. Let N be the number of cubic centimetres of iodine added. The iodine contained in the test will correspond to 20 N cm³ of liquid titrated, and the test will consequently contain $20 - N$ cm³ of oxygen.

The method is simple, rapid and exact. It is delicate in application in the case in which the water contains traces only of oxygen, on account of the avidity of hydrate of manganese, which absorbs instantaneously the oxygen of any bubble of air coming into contact with the water. It is necessary to ascertain that all the reagents are pure, and particularly to make a blank test of the hydrochloric acid on iodide of potassium.

Removal of the gases. The elimination of the oxygen dissolved in water may be effected either by physical methods, or by chemical methods, or by the two combined. The principal physical methods to be mentioned are: agitation of the water; the action of temperature; and the action of vacuum. These three methods may be used either separately, or in the majority of cases in combination, in order to obtain the elimination of the gases from the water. Of the chemical methods the only one to be considered is the action of highly oxidisable iron, which absorbs the oxygen from the water before entering the boiler.

Removal of the gases by physical methods. Agitation of the water is used in various appliances in the form of fine spraying or circulation through channels of varying section, or again by the action of points and roughnesses on the current of water. These can only have the effect of preventing supersaturation at the particular temperature and pressure. Consequently they can have no effect beyond that of the action of temperature combined with a vacuum. Raising the temperature diminishes the solubility of the gases. But even if the temperature of the water is raised to 100° C., the solubility cannot be neglected; solutions containing about 2 cm³ per litre can be obtained at atmospheric pressure. The operation of heating, however, produces by itself a great improvement in the condition of the water entering the boiler, for there is no longer such a large release of air when the feed-water comes into contact with the water at a higher temperature in the boiler.

A vacuum would give better results still if it could be carried out at a very high temperature, the solubility of air being proportional to the pressure, according to Henry's law, as has been already stated. But the vacuum, particularly when the water is hot and attains 80° C., cannot be very high, for otherwise the water would boil. At 80° C. it is not possible to fall below about 0·5 atmosphere, and at 60° C. more than 0·8 atmosphere.

If the three methods mentioned are used in combination, that is to say, if the water is submitted

to the combined action of temperature and a vacuum, and supersaturation is prevented by mechanical action, the results shown in the following diagram will be obtained, Fig. 12.

This curve has been prepared and published by a large German company making an apparatus, which uses the three methods simultaneously. It appears to be, if examined carefully, favourable to the use of these methods. Notwithstanding this, the firm is obliged to state: "There are still some milligrammes of oxygen that cannot be removed by a very high vacuum and by reheating the feed-water, but they have no aggressive action; the danger arises alone from a rich supply of oxygen."

The action of iron on dissolved air in the feed-water enables even the last traces of oxygen to be removed and water to be obtained absolutely free from oxygen. This proves that even the "last milligrammes" of oxygen are capable of causing rust and corrosion. Nevertheless, the physical operation of removing gases gives very satisfactory results, because it diminishes the percentage of oxygen in the feed-water and slows down the corrosion. But the use of a vacuum appears very difficult in appliances of large size, as it requires a very keen supervision at all points; and when leakage occurs, there is a churning effect produced in the water by the entry of the external air which, notwithstanding the decrease of pressure caused by the air temperature, produces the opposite result to that which is desired. A leakage of this kind is hard to locate, and for this reason it is always possible that such leakages may exist.

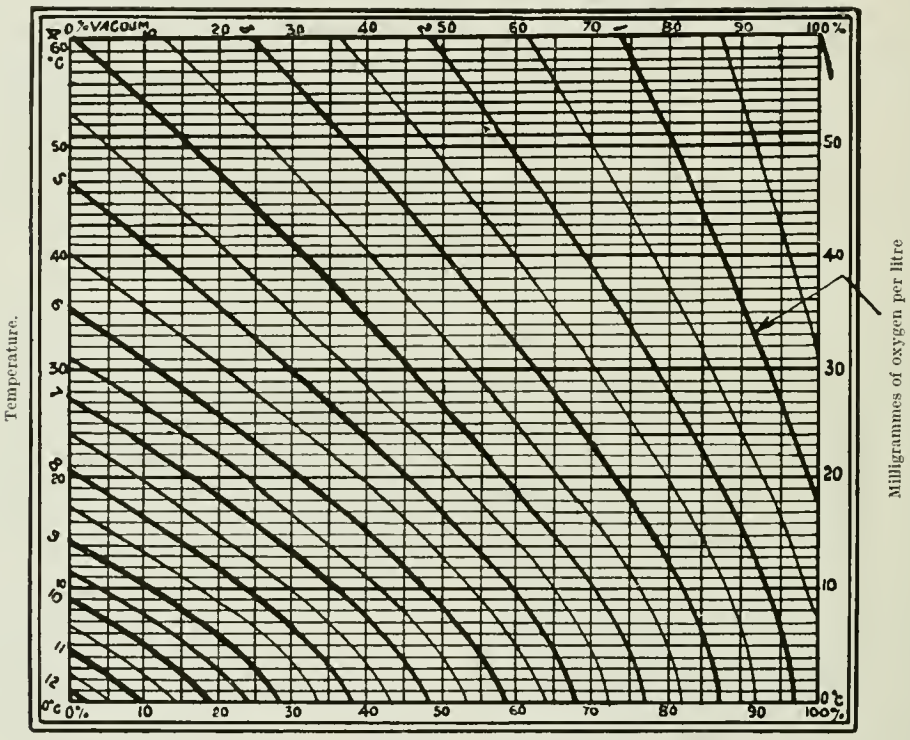


FIG. 12.

Absorption of oxygen in water at different temperatures and vacua.

Numerous appliances have been constructed in England, America, and in Germany for effecting physical removal of the gases. The principal ones in use are the following:—

(A) *Thermal and mechanical removal of gases.*—All feed reheaters open to the air produce a removal of gases in so far as they diminish the solubility of air in water. Those in which the water flows in contact with steam in reservoirs that are not tight are those which best perform the operation of the removal of gases by heat alone. Those that reheat the water in closed receptacles, *e.g.*, in tubular economisers, remove the gases to a great extent from the water, but introduce gases into the boiler when it becomes necessary that the gases that have been removed are allowed to escape. Among these appliances those most in use are superheaters by Cochrane and Webster, and Weir, etc.

The apparatus of Chevalet, by its construction as a superheater with plate surfaces, differs slightly from the preceding appliances, and in this the steam bubbles through the water. All the "heating nozzles" of apparatus in which water is subjected to mechanical action and to the action of heat come within this class.

These appliances are accessories in the operation of removing the gases; there is one which can lay claim to the production of a better result, and that is the Aerex apparatus, in which the water loses its gases in being divided over two rubbing surfaces. The gases reunite and are removed by a valve when they have attained a sufficient quantity.

The Sigemon Schmidt gas remover adopts a principle similar to that of partitions with baffles. The two appliances are placed on the delivery; they have more effect when the water is hot, but, on the other hand, they suffer some reduction in their action in consequence of the pressure on the feed-water, the pressure assisting the solution of the gases.

To eliminate gases by heat the firm of Balke, in Germany, combines an arrangement for reheating

the water by steam with extraction of the gases evolved in an appliance in which the water is subject to forced circulation, and the air removed is exhausted by an arrangement of valves at the top of the apparatus. The water freed from gas is kept in contact with a layer of inert gas at the lower part of the apparatus. This somewhat complicated installation does not appear to have met with much success.

(*B*) *Vacuum appliances for removing gases.*—Some firms have constructed apparatus using a vacuum for removing gases from water. Most of these are schemes of apparatus which have but rarely been developed into practical shape; such are the arrangements of Meihof worked by Halvor Breda and the Griesheim Elektron Lauta. The "Deutsche Sanitäts Werke" of Frankfort have constructed apparatus which is of considerable interest.

The Meihof, applied at Swinemunde, acts by passing the water from which the gases are to be removed through a siphon arrangement. If the branches of the siphon are of sufficient length, a vacuum is produced at the upper part, due to the suction in the descending branch. This vacuum causes the release of gases dissolved in the water at the top of the siphon. The principle is very simple and requires no mechanical arrangement; the water from which the gas has been removed is returned to the level of the water supply. The vacuum obtained in such an arrangement is not very high; it is limited by the destruction of the vacuum in the siphon by accumulation of gases. When these gases are removed, the vacuum disappears; this removal is produced by valves arranged on the particular part of the siphon operated at the time when the water returns for priming the siphon. In the apparatus constructed at Swinemunde for the removal of gases, the maximum vacuum corresponded to 5 metres (16 feet) of water, which is not a high degree of vacuum.

The apparatus of the Griesheim Elektron Lauta is of very simple construction, consisting of a reservoir in which the temperature of the water is raised by means of steam. A vacuum is created by the heated water and the gases that accumulate are extracted by means of a pump.

The apparatus made by the Deutsche Sanitäts Werke is largely in use and is of very original design. In principle it consists of a tube with numerous contractions, to the upper part of which a vacuum pump is attached. At the base of this tube, which is immersed in the water from which the gases are to be removed, there is an adjustable air suction-pipe. Doubtless this air passes over an oxidisable filter in order that the oxygen may be removed from it. As a result of the vacuum, the water and air that have been filtered riso in this column; there is a vigorous shaking of the liquid due to the passage of the gas and to the contractions. This violent movement in conjunction with the vacuum causes the release of the gases dissolved in the water, and these gases are drawn into the upper part of the apparatus by the vacuum pump. It is certain that different conditions prevent a high vacuum from being obtained, in particular the shaking by use of gases and the height of the apparatus, which is far from attaining the dimensions required by the barometer. For this reason it is said "that there are still a few milligrammes of oxygen remaining in the water"; nevertheless, this apparatus certainly removes the oxygen to a great extent from the water that has passed through it, and it contains at its maximum the percentage corresponding to saturation at the pressure obtained in the column and at the temperature of the water. This apparatus appears to be in wide use.

Chemical removal of gases.—All chemical methods of removal of gases in actual use are based on the same principle. They consist in causing the water that it is desired to free from oxygen to pass over iron. If the iron has a sufficiently large surface, if its chemical composition is carefully selected in order that it may be very easily oxidisable, and, lastly, if the water is circulated sufficiently slowly for the oxidising action to be complete, the chemical removal of the gases enables all the oxygen contained in the water to be eliminated; in fact, the oxygen may be combined with iron in the presence of water so as to give different hydrates, of which the richest is rust:

$$2Fe+3O+3H_2O=2Fe(OH)_3.$$

This reaction is practically quantitative; it requires more or less time according to the temperature at which it takes place, but it can always, whatever be the temperature, effect a sufficiently complete removal of the gases, if the iron is largely in excess in regard to the oxygen, so that nothing but minute traces of gases remain in the water.

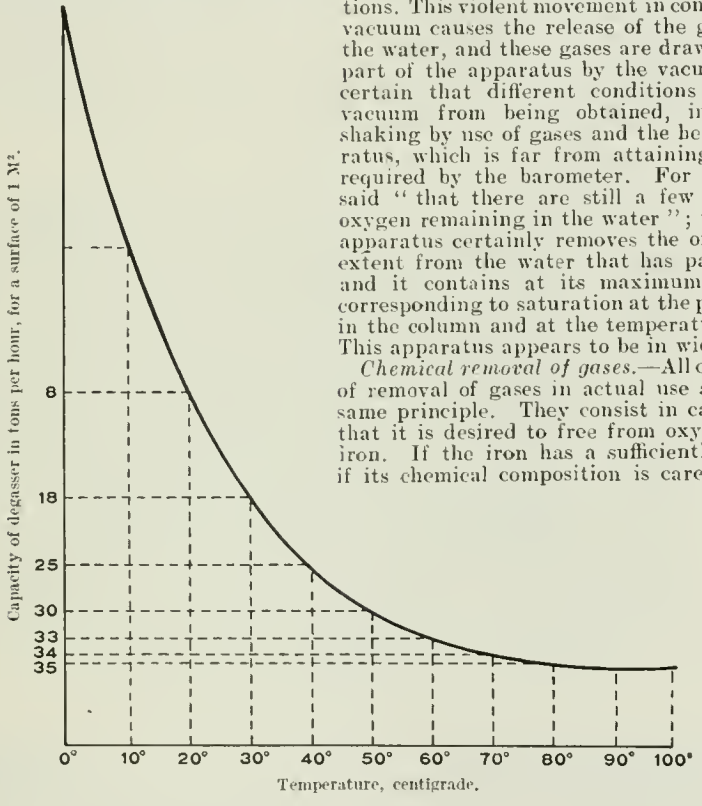


FIG. 13.

Output of a filter given as a function of the temperature.

For the same quantity of iron turnings, the quantity of water from which the gases are removed is proportional to the speed of the reaction, and consequently proportional to the temperature. The output of a filter is given as a function of the temperature by the following curve, Fig. 13. If the velocity is caused to vary in a given filter, it will be seen that it cannot yield more than a certain limited quantity if it is desired that the removal of the gases should be efficient.

The output is also proportional to the quantity of turnings in contact with the water; this quantity, moreover, cannot be increased indefinitely because the size of the apparatus would require to be sufficiently reduced so as not to be an obstacle to its introduction into a boiler room.

Another factor intervenes in the working of these appliances; it is the facility with which the metal used becomes oxidised. Practice shows that very impure iron rusts very rapidly. Laboratory experiments have enabled the author to find a catalytic agent of great importance, namely, manganese. The presence of manganese hastens the oxidation of the metal; nevertheless, if this action shows itself in the case of small percentages corresponding to the manganese steels, it is not so well marked in the case of high percentages. A metal which is rich in manganese is cast-iron, and cast-iron is much less oxidisable than steel. The physical and chemical composition of the metal has therefore a great influence.

The catalytic action of manganese may be represented by the following curve, the metals under consideration being very closely allied in composition and physical properties and differing only by their percentage of manganese, Fig. 14.

To sum up, it is seen that from the chemical point of view, the removal of gases by iron depends: on the surface of the metal, on the temperature of the water, on the speed at which it passes over the metal, and, finally, on the composition of the iron.

To obtain a maximum surface with a certain quantity of iron it must be used in a finely subdivided form, preferably in turnings. There is then a physical removal of the gases at atmospheric pressure, which reduces the gases in the water to the point of saturation for air at the temperature and under the pressure in question. This action is of great advantage, and reduces the amount of turnings required to a considerable extent.

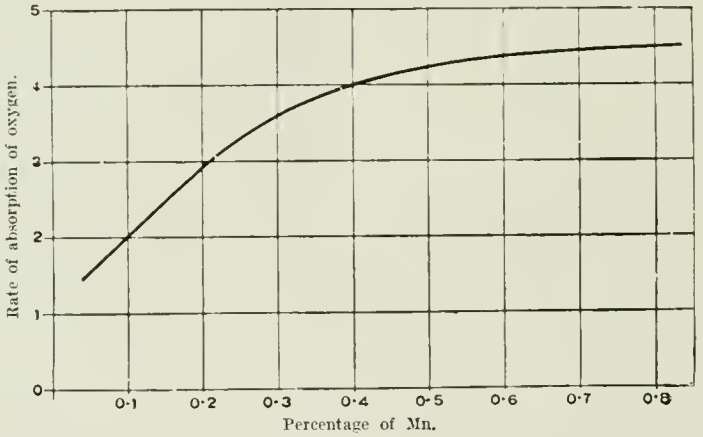
In commercial appliances, the following phenomenon may occur: When the coating of rust becomes very thick, contact of oxygen with the iron is no longer good, and the chemical removal of the gases may be considerably slower. Notwithstanding this, under the worst conditions, a water leaving such apparatus may be found to contain less than 0·5 cm³ of oxygen per litre. There are some other appliances working on the same principle as the German degassers constructed by the firms of Hulsmeyer and Seiffert, and an American apparatus due to Professor W. H. Walker.

The Hulsmeyer apparatus consists of a column of iron turnings distributed in baskets of perforated sheet-iron, which allow it to be distributed equally. This column of iron turnings ends in a filter which retains any particles of iron-oxide that may become detached. At the top of the apparatus there is fitted an automatic float-operated valve for the relief of the gases released by physical action. The water circulates from the bottom to the top of this very simple apparatus. The Hulsmeyer apparatus may be included as either falling under the suction type or the pressure type.

The Seiffert apparatus is constructed on the same principle, but comprises a reheater. Starting with the fact that heat encourages physical and chemical removal of the gases, Seiffert filled the tubes with a tubular superheater filled with iron shavings. The apparatus is of the most simple kind, and is, perhaps, very effective, but the replacement of the iron shavings in the tube cannot be a very easy matter.

Prof. W. H. Walker proposes an apparatus for removing the gases from hot water, consisting of a receptacle filled with perforated plates one above the other. This apparatus he styles a "deactivator." The only point that appears to have been overlooked is the absence of a valve for allowing the gases released in the apparatus by its mechanical action under the influence of the circulation of water between the plates.

To avoid the slowing of the oxidising process by the rust deposit on the iron, the Kestner Co. has designed an apparatus based on the following: If an apparatus for removing gases by means of iron is left with the iron rusty, it is found that after a day of rest the rust, which was red, has become an oxide of a greenish-black colour. This transformation is due to the reduction of the ferric



The rates of absorption of oxygen are expressed in equal proportions for the same velocity of flow of water through the apparatus.

FIG. 14.

Velocity of absorption of oxygen for degasser.

hydrate under the influence of the iron; a saline hydrate of the oxide and a ferrous oxide is obtained. The following is the reaction:—

$$2Fe(OH)_3+Fe+2H_2O=Fe_3(OH)_8+2H.$$
$$2Fe(OH)_3+Fe=3Fe(OH)_2.$$

This reaction takes place in the middle of the reducing agent, that is, in the presence of iron and in the absence of oxygen. The saline oxy-hydrate formed is easily removed from the iron by washing, and, moreover, it is capable of further absorbing oxygen in becoming ferric-hydrate. In order that the apparatus for removing the gases should retain its working speed it is necessary that it should be put alternately in and out of circuit, the two appliances in work retaining the power of the iron for absorption until the latter has been completely oxidised. The Kestner apparatus, Fig. 15, is based upon this principle, and consists of a column of turnings of sufficient quantity for half only to be in action, and to allow that the water shall have lost these gases on arriving at half the flow of the water can be inverted at regular intervals. Like the other filters using iron turnings it may claim completely to eliminate the oxygen from the water with greater certainty. The consumption of turnings and the speed of the absorption of oxygen are approximately regular, and the arrangement for allowing steam to be admitted at the base of the apparatus allows it to be cleaned by blowing steam through and removing any rust which may become detached from the iron on which it was produced.

The consumption of iron turnings in the apparatus for removing gases is very small; according to the percentage of oxygen contained in the water it varies from one to three grammes per cubic metre (per ton) of water, which reduces the cost of the removal of gases to a negligible figure. The iron consumed in the gas-removing apparatus corresponds to the same weight of boiler-plate which has been saved from destruction, and the difference in first cost between the two materials destroyed forms a very great saving.

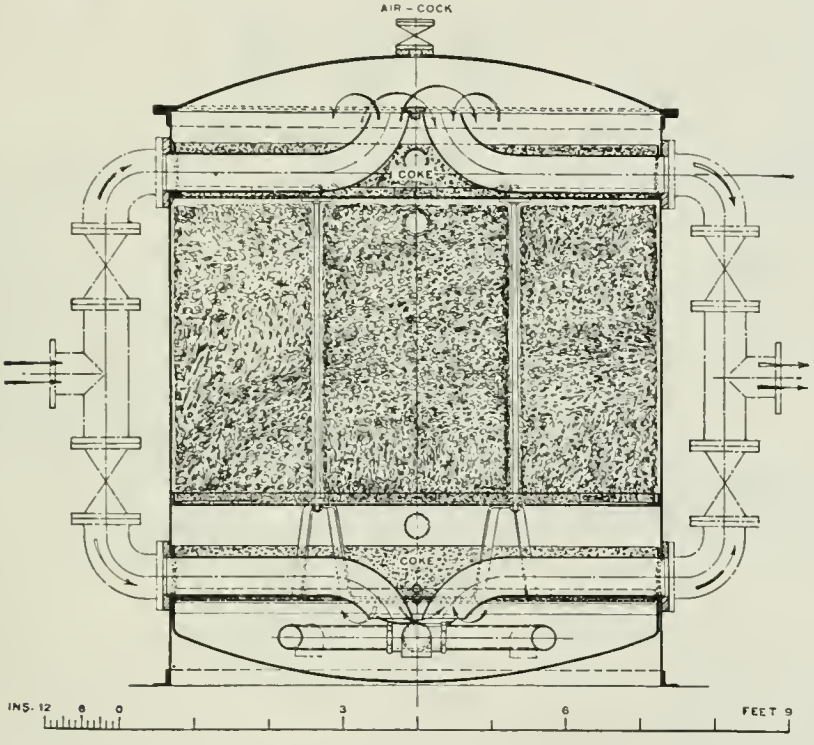


FIG. 15.

Arrangement of degasser. (Kestner).

height. The second portion of the iron being in contact with this deoxidising agent, gives rise to rapid reduction, its rust changing to the condition of a suboxide. At the end of the day the direction of flow is reversed. The water then passes over the iron, which has become a suboxide, which is then changed to a peroxide, and arrives free from gases on the surface (rusted the day before), which in turn is reduced. Thus each half of the apparatus works alternately by inversion of the direction of circulation of the water, and the removal of the gases becomes continuous and absolutely regular.

The Kestner apparatus consists of a column of iron turnings placed between two filters, with an intake and outlet for the water at each end of the apparatus in such manner that the direction of

Whatever may be the method employed for removing the gases it involves a circuit of water closed from atmospheric contact if it is to give a large output. The water which has been freed from gas can become saturated very rapidly by air. It is therefore advisable that the tanks in which the water may be retained should be as well enclosed as possible, and that on the whole of the pipe system there should be a small positive pressure. A head of a few centimetres of water is sufficient to prevent the entry of air which might happen accidentally through bad joints or accidental circumstances. In such a pipe system the only place at which the entry of air may be possible is the condenser in which the vacuum is produced. The tightness of the condenser should therefore be very

carefully tested. Moreover, to avoid the effect due to any air entering by accident, which may always occur, it is essential that the water should pass through the gas remover immediately before entering the boiler, even though this water should result from condensation, and though it may only contain a small quantity of oxygen. Iron turnings are only consumed according to the percentage of oxygen in the water, and water free from gas does not change this; consequently it does not necessitate expense by being passed through the degasser. The degasser so placed protects the boiler and the economiser from any attack by the gases, and acts as a doorkeeper to prevent the entrance of anything undesirable.

The study of continuous blowing-down, on the one hand, and that of degassing on the other hand, have led to the conception of a complete

The degassing of the water to be purified before it passes into the distilling apparatus.

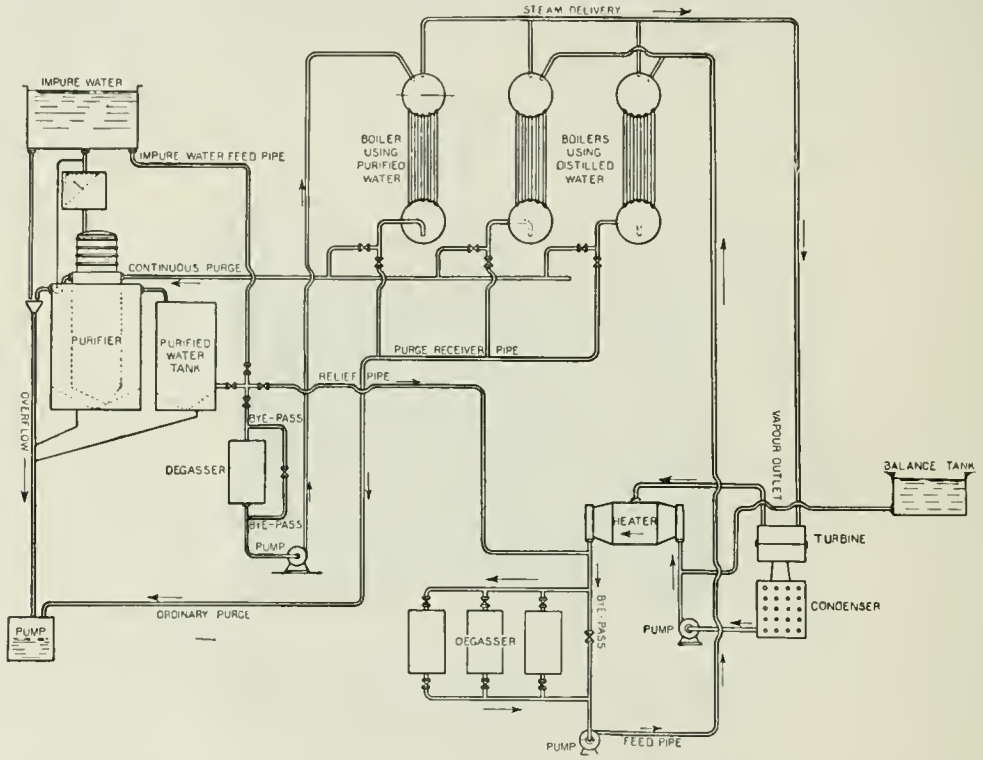
There should be no point on the circuit of the purified water in common with any on the circuit of the distilled water.

The only appliances that may be common to the boilers are the blow-off manifolds and the steam mains.

It will be seen that the scheme of this system of water circulation purification and degassing rests on a very broad basis, and that the distilling apparatus may equally well be an evaporator or a boiler. Each has its respective advantages.

For a water circulating, purifying and degassing plant the following system is proposed, Fig. 16.

One of the boilers is to evaporate the purified water coming from the purifier. It will supply steam that will go with that generated by the other



Scheme for the purification of make-up water by continuous blow-down and degassing of all water before discharge to boilers. One boiler operates on purified make-up water; the remainder using distilled water only.

FIG. 16.

Diagram of degassing, purifying, and distilling plant, using purified and distilled water. (Kestner).

circulation system for a central station using distilled water. The main guiding principles of this idea are as follows:—

The ability to use any one boiler as a distilling apparatus and to feed the other boilers with the water distilled in this boiler.

The blowing-down of a large quantity of water from the boiler that is performing the function of distilling; the blowing-off of only a small quantity of water from the boilers using distilled water and the conveying into the distilling boiler of the water so blown-down.

The protecting of the condensed water from all contact with the air by means of a closed circuit; the degassing of the condensed water before its passage through the economisers and the boilers.

From the moment at which the water is degassed the keeping of it enclosed so that no entry of air is possible.

two boilers to the steam main. The steam will pass through the turbines, it will be condensed, and it will re-enter the boilers as distilled water after it has passed through the degassing apparatus.

All the boilers are to be blown-down, the boilers using distilled water to the extent of, say, 2½ per cent., and that performing the distillation of the water to the amount of, say, 10 per cent., and all of these amounts that have been blown-down are to pass collectively through the purifier, in which they will be subjected to the same treatment as the ordinary "make-up" feed-water, and the impurity is to be measured by the testing apparatus. The purified water is to be returned to the particular boiler performing the distilling after it has passed through a special degassing appliance.

The boiler containing the water that is to be purified will thus furnish the supply to the boilers working with distilled water in the form of steam;

there will be no common portion between the circuit of the purified water and that of the distilled water. It will merely be necessary to provide an overflow reservoir to enable any excess of purified water to be reintroduced into the circuit if such excess should be produced.

All the boilers can be fed either with purified water or with distilled water, which enables any desired boiler to be used for the work of distilling.

Such a circuit will ensure that a central station will have the best possible thermal balance-sheet, and moreover a longer life for the boilers, which the process protects from formation of scale and corrosion.

It is obvious that a similar scheme can be arranged for the distillation by means of an evaporator, which under certain circumstances offers advantages. Such an arrangement is illustrated in Fig. 17.

The paper is illustrated by 17 Figs. in the letterpress.

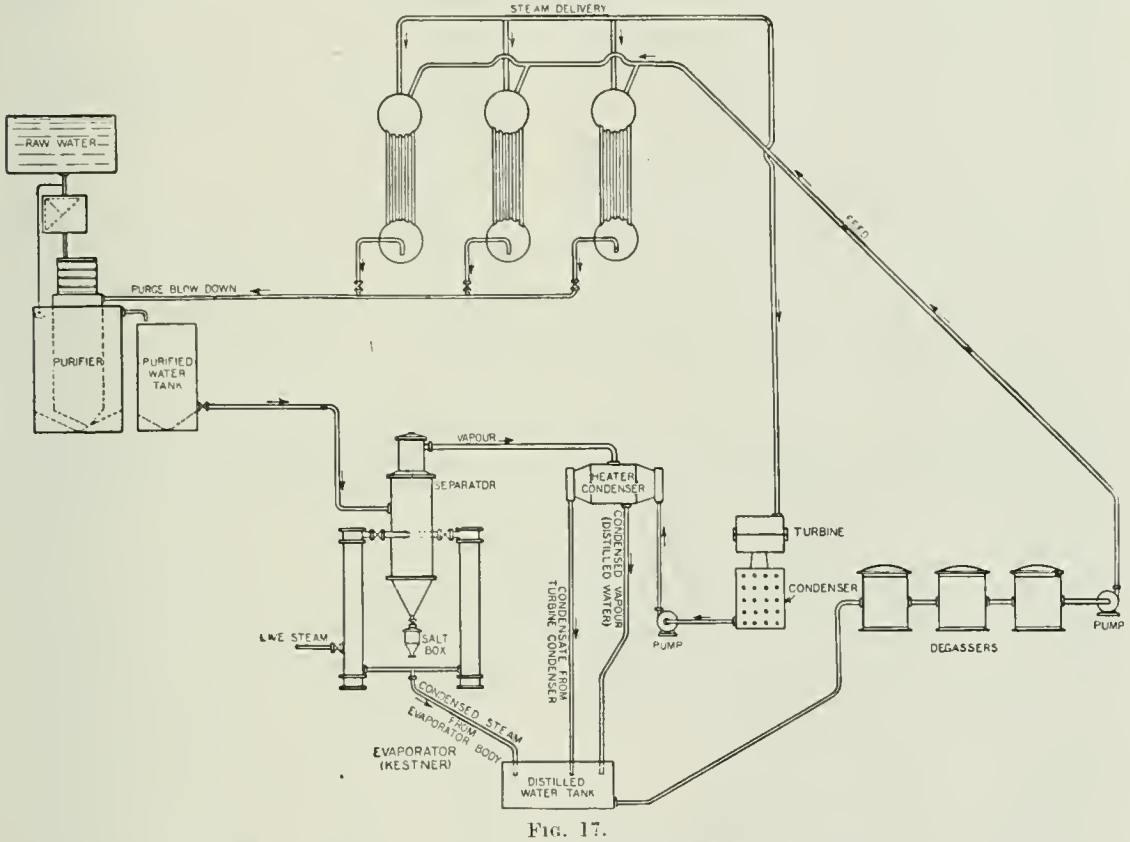


FIG. 17.

Diagram of degassing, purifying, and distilling plant, using purified and distilled water. (Kestner).

DISCUSSION.

The PRESIDENT said that in view of the applause with which the paper had been greeted it was hardly necessary to propose a formal vote of thanks, and asked the author to accept the thanks of the meeting. The paper brought very forcibly to his mind the necessity of an *entente* between mechanical engineers and chemists. Chemists indicated what was wanted in regard to various operations, and it then remained for the mechanical engineer to devise the apparatus whereby those operations could be carried out.

The resolution of thanks was then put and carried with acclamation.

Sir WILLIAM POPE, K.B.E., F.R.S. (President of the Society of Chemical Industry), said the pleasant duty fell to him, on behalf of the Society of Chemical Industry, to welcome the author to the joint meeting of their two great societies. The profession of chemical engineering had now become a well established one, and amongst its numerous members there were, as in every other profession, a few who stood out in that their work always bore the stamp of genius. They were accustomed to look for that mark of originality in everything the author did. They had seen it in the past; they had discerned it in his paper that evening, and they looked forward to seeing it again for many long years to come in his future work. Their recognition of the author as an artist in a great profession was evidenced by the fact that the Society of Chemical Industry had conferred upon him the highest honour in their gift, namely, the Medal of the Society, and he was the only Frenchman who had received that award up to the present.

The author had always been a great advocate of the necessity for closer co-operation between his own nation and this country, and it was because of his belief in that necessity that he had put himself to the inconvenience of coming to London to address the joint meeting of the institutions. But he thought on the present occasion something more than that might be said. Captain Sankey had remarked on the necessity for more mutual effort between the chemist and the engineer, and in the present joint meeting there was to be seen, he hoped, the inauguration of a period of closer co-operation between the two great subjects with which they

dealt. The alleviation of many of the troubles from which the world was now suffering depended upon progress in engineering, in chemistry, and in chemical engineering. It was no part of his task on the present occasion to open a technical discussion on the subject before the meeting. There were many present who had a very wide practical acquaintance with the subject of the paper, and he would therefore leave the discussion of the important points which had been raised to those who were better able to deal with them.

Professor J. W. Hinchley, in opening the discussion, said the author had been labouring at the subject a long time, and he believed he had succeeded in evolving a practical solution to a very difficult problem, namely, that of freeing water and steam raised from it from the corrosive oxygen and carbon dioxide usually present. The fact that ferric hydrate in contact with iron was slowly reduced to the ferrous state, and was again capable of performing the function of an oxidizer, was a remarkable chemical fact to be used in solving the practical problem. The absence of corrosion in the boiler would be not less important than its absence in other parts of the plant through the freedom from oxygen of the steam. The presence of air in steam was one of the bugbears of the chemical engineer. He did not believe the mechanical engineer worried about it, but he certainly had great evidence of it in the damage that was done to condenser tubes and other parts of his plant. He was very interested in the fact that the author had found that cast-iron containing manganese was an extremely active rusting agent. He believed cold blast cast-iron liners were usually stated to be used in ordinary cylinders of steam-engines, but there was little doubt that many of those liners were cast from metal made in an ordinary cupola by the addition of steel containing appreciable amounts of manganese, and it was quite possible that much of the corrosion that was obtained in those cylinders might be due to the oxygen in the steam and the presence of manganese in the cast-iron. He believed manganese was a notorious agent for producing segregation, especially of sulphur as manganese sulphide. He would like to know from the author whether he determined the percentage of sulphur at the same time that he determined the manganese, and whether the segregation of manganese had anything to do with those characteristic properties which he found, or whether it was merely manganese in solution in the cast-iron or equally distributed through it. He would also like the author to say whether he found that the amount of graphitic carbon in the grey cast-iron which he used had any great influence.

Mr. Arthur Spyer said it was difficult for him to criticize the paper, because he was so absolutely in agreement with the theories the author had enunciated, especially in regard to the necessity of degassing feed-water. The author had pointed out that carbonic acid gas and oxygen, when they were present in the feed-water, resulted in a recuperative action, because ferrous carbonate was first formed, then in the presence of oxygen this was transformed into ferric carbonate, which, being a very unstable body, again resolved itself into oxide of iron and carbonic acid gas, so that, if the carbonic acid gas was not got rid of, the action was continuous and recurrent. The author had spoken at great length on the so-called chemical method of degassing, and he was very much interested to hear the propositions he made in reference to the use of what might be called the steel-shaving method, because until quite recently the results of degassing with that method of treatment had not been altogether consistent; in fact, in many cases it was known they had been unsatisfactory. There seemed to be several indications, however, that with the modifications suggested by the author those difficulties might be overcome.

Another point raised was the use of evaporators. The vapour produced in ordinary evaporators frequently contained carbonic acid gas, oxygen, and traces of copper salts. If these substances obtained access to the boiler-water, they reproduced the gas difficulty and hence the necessity of getting rid of the gases. The most agreeable waters for drinking purposes were usually impregnated with carbonic acid and oxygen, and whilst exceedingly pleasant to drink, they were exceedingly bad for boilers.

With regard to the suggested method of a continuous blowing out, he did not wish to be hypercritical in any way, but in a well-managed station at the present day, with the methods which were adopted of getting rid of solid matter in the feed-water, the amount of blowing out from the boilers should be very small. It was rather doubtful whether it would pay commercially to install somewhat costly apparatus as described in the paper, unless very definite evidence were forthcoming of the gain to be obtained, because, after all, engineers unfortunately had to consider whether a particular apparatus paid or not.

In dealing with the subject of corrosion, the author had referred to galvanic action. In his own experience he had never yet seen a case in which he was convinced that galvanic action of itself had been the cause of corrosion. The wear of the zinc blocks which were frequently fitted in boilers was more probably due to ordinary oxidation than to galvanic action. He was absolutely in agreement with the author with regard to the beneficial effects of heating the feed-water before subjecting it to the discharge pressure; it undoubtedly helped to get rid of gas, and facilitated the action on the water of any chemicals which might be introduced. It reduced the solubility of the salts at high temperature, and therefore enabled a great proportion of the salts to be got rid of before they went into the boiler. It should be the object of engineers and chemists first of all to take care that no air got into the feed-water; in the second place, to take all possible steps to prevent the access of air to the feed-water subsequently; and thirdly, to heat it as much as possible so that any gases which were present might be eliminated so far as practicable.

Mr. R. W. Bailey said he was sure that builders of steam-turbines would be indebted to the author for his valuable paper; first, because it emphasized the need of removing oxygen from boiler feed-water; and secondly, it might be inferred that, if it was not removed, serious corrosion of the plant might result. Corrosion in a steam-turbine was a much more serious matter than in a boiler; and there was no doubt whatever that oxygen was responsible in many cases for blading decay. Although materials specially resistant to corrosion were employed in the construction of steam-turbines—for example, 5 per cent. nickel steel, high chromium steel, and special alloys—corrosion of blading might occur unless proper attention were paid to the feed-water. The firm with which he was engaged, namely, the Metropolitan-Vickers Electrical Co., had experienced cases of blading corrosion, and its research department had investigated the problem of water treatment with a view to its prevention.

The action of an iron deoxidizer of the type described by the author was investigated, and the results obtained confirmed his remarks regarding its effectiveness. At first it was thought that iron turnings would meet ordinary requirements, but the idea was abandoned as it was feared that the water would traverse the mass in lanes, and uncertain and irregular action would result. He would like to ask the author whether trouble of this kind was experienced with iron turnings. Attention was therefore directed to the use of expanded sheet

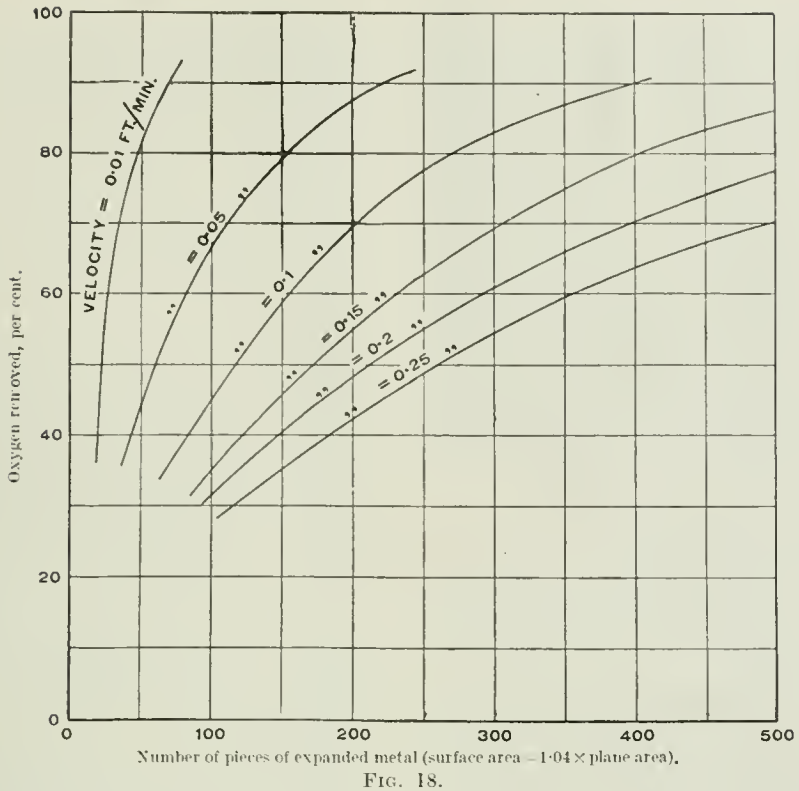


FIG. 18.

steel, and Fig. 18, based upon experiments made with a small deoxidiser, showed the relation between the amount of iron, rate of flow, and oxygen removed as a percentage of that initially present. The action of such a deoxidizer was also investigated from a theoretical standpoint, in order that rational formulæ might be employed which could be used with some degree of confidence outside the range of

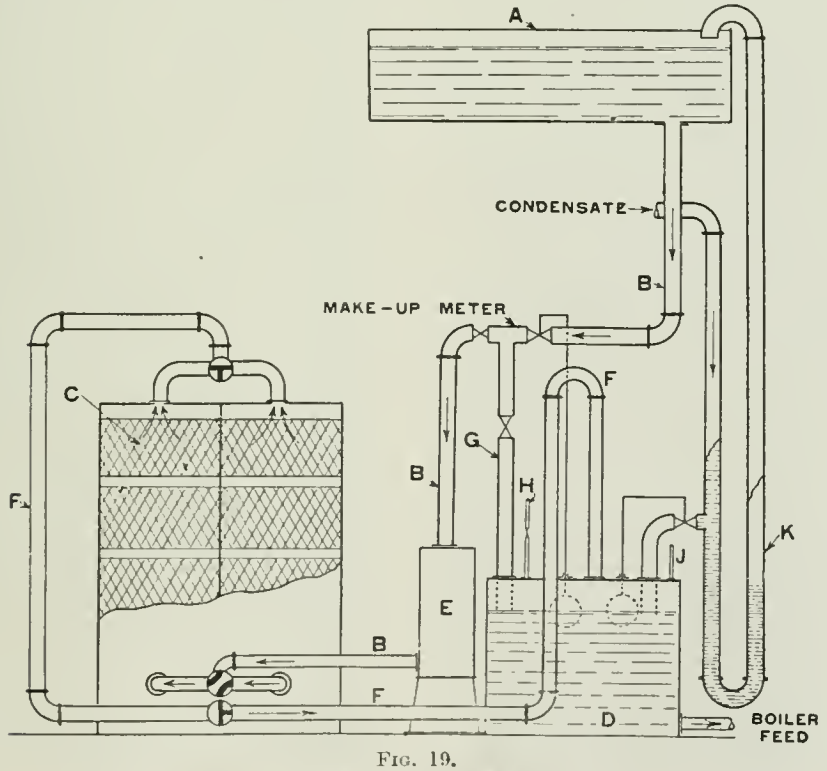


FIG. 19.

Diagram of feed-water de-oxidation plant. (Metropolitan-Vickers Elec. Co.).

the experiments. This was done in Fig. 18, which gave values for cases up to 500 sheets, although the experiments were only conducted over one-tenth of this range. It was found that the rate at which the iron absorbed oxygen was rather more than proportional to the concentration of oxygen (this might not be absolute, but might be really due to a more extended surface being offered where oxidation was most active), and increasing the velocity of flow increased the rate of absorption at the iron; but, as the quantity entering was proportional to the velocity, the percentage of oxygen removed from the water decreased with increasing rate of flow, as would be expected. The curves showed that the increase in efficiency with decreased velocity was appreciable. A rational formula taking into closer account the effect of concentration and velocity was not convenient for practical purposes, and the following approximate formula, which would satisfy most practical cases and was a basis of Fig. 18, might be given for the case where the water temperature was 15° C.

$$\mathrm{Log}_{10}\frac{100}{100-P} = \frac{3{\cdot}1\ S\ N}{100+12500\ V} + 0{\cdot}04$$

Where:—P = Percentage of oxygen removed.
V = Velocity of flow in feet per minute, based upon the gross cross-sectional area.
N = Number of sheets of expanded sheet steel.
S = Ratio :— Area of actual iron surface / Plane area of sheet (one side)

The influence of temperature in increasing the rate of oxidation was most marked. Their information on this point was not very extensive, but it appeared to support the rough rule that it doubled for every 10° C. increase in temperature. He would like to ask the author whether such a relation agreed with the curve exhibited by him.

He was most interested in the alternating flow arrangement developed by the author in order to take advantage of a regenerative action. His firm had developed a similar arrangement for a different reason, namely, that the rate of corrosion was so much greater at the inlet end that, to secure something like uniform oxidation of the iron throughout the deoxidiser, it was necessary to employ the alternating flow principle. It should be mentioned that the rate of oxidation at different positions in a deoxidizer of the kind considered was given approximately in Fig. 18 by the ordinates of the different curves measured from the 100 per cent. line.

The Metropolitan-Vickers Electrical Co. did not make deoxidizers, but they had looked into the question of their action and design in order that, where necessary, recommendations could be made to customers in connexion with the steam-turbines supplied. Fig. 19 showed diagrammatically a scheme recommended in the case of a large electric power-station in this country where corrosion had been traced principally to dissolved oxygen in the feed-water. Only the make-up water was passed through the deoxidiser, and this flowed under gravity from the storage tank A, via the pipe B and thermostat controlled heater E, to the deoxidiser C, its rate of flow being controlled by a float in the hot-well D. The valve arrangements for reversing the direction of flow and for permitting one side to be opened up while the other was in operation, thus enabling replacement of the expanded metal to be carried out without stopping the plant, as also the arrangement for shutting down the deoxidiser completely, would be understood from the figure. To prevent subsequent aeration of the make-up and of the condensate, the hot-well D was steam-sealed, exhaust steam from auxiliaries being led in at H and its supply adjusted so that a small escape took place at the vent-pipe J. To prevent aeration of the condensate and at the same time permit a simple delivery to the overhead storage-tank when the level in the hot-well closed off its flow thereto, an inverted siphon K was employed and the branch to the hot-well was "teed" off the inlet tube as shown. A column of "dead" water existed below this connexion, and thus sealed the condensate passing to the hot-well from the atmosphere. The delivery pipe from the deoxidiser was carried up as shown at F, in order to keep the upper part of the deoxidiser under sufficient pressure to enable a snifting valve to release any gas that might accumulate when heating was employed.

Professor F. G. Donnan, F.R.S., said he remembered that some years ago the question of the degassing of the water in the Calgoorlie water supply main, which had a very great length, arose, and he understood it was done by spraying at a reduced pressure. That was a very important question in connexion with conducting water over long distances and for general questions of municipal supply. It would be of interest if the author could state how that method could be developed for general supply purposes. It was quite impossible to use a chemical method on a very large scale like that, and it would therefore be of interest to know if it was possible to use the physical method successfully. He thought that spraying by nozzles at a sufficiently low pressure-vacuum would not remove the oxygen sufficiently, but that was not a matter on which he possessed enough knowledge to give a valuable opinion.

With regard to the author's scheme of a boiler acting as a water distiller, he remembered inspecting in South America a boiler system which worked in very nearly the same way. One boiler took the make-up feed, which was softened in the usual way, and acted as a water distiller to the other boilers. The condensed water came back, and there was a partial attempt at systematic blowing down, but it was not very great and was not particularly necessary. The boilers were mostly water-tube boilers working at 200 lb. pressure, and the water distiller was a Lancashire boiler working at about 50 lb. pressure.

Professor J. S. S. Brame said he had been carrying out a good deal of work in connexion with boiler corrosion, and he was quite convinced that the question of oxygen in the feed-water was one of the utmost importance. The author considered that the action of water and oxygen first produced ferric hydroxide, a similar action to that which took place in corrosion at normal temperatures. He (the speaker) was not sure, however, that the actions were similar in a boiler where the temperature was somewhere of the order of 200° C. In the deoxidising of water by iron, the interesting point was the reduction of the Fe_2O_3 in contact with the iron in the absence of oxygen. At the point of entry the Fe_2O_3 existed as a maximum, but by passing the water in the other direction a dark greenish-black compound was obtained. It was an action which had been very largely overlooked, although it was mentioned in the old text-books; for instance, a very interesting account of that action would be found in Gmelin's Chemistry (vol. v. p. 185: 1851). The author had postulated the formation of a hydrated magnetic oxide $Fe_3(OH)_8$. He (Professor Brame) would like to know whether there was any basis of analysis for such a formula. It was most difficult to get consistent results for the analysis of any of those products of oxidation except the fully hydrated oxide. From most forms of rust a considerable proportion of a magnetic oxide could be separated by grinding them up to a very fine state of division and flowing them by means of water past a powerful magnet. One was so accustomed to look

upon it that the formation of ordinary Fe_3O_4, either as Fe_3O_4 or in the hydrated form was only produced under special conditions that it was somewhat surprising to find that, if a plate of iron was left just awash in water, so that plenty of oxygen could get through, the red rust formed first of all, and that as the mass was built up there was formed underneath a darkish green or almost black compound which was strongly magnetic. Those were all reactions which related to the question of separating the oxygen from the water by the iron process, and in addition they must play a very important part in the whole question of corrosion. The solution of the problem lay very largely in finding out first of all what types of action were involved.

The author had referred to magnesium chloride. It had had a lot of sins to bear and a lot of burdens to carry, but he was not sure that it was such a sinner as it had been made out to be. If the whole of the magnesium chloride remained in solution, he thought it was very doubtful whether that action of hydrolysis produced the hydrate and free acid actually did take place, but it had been shown by Hof that the development of free acid only became an appreciable reaction when the magnesium chloride was left as a deposit on the side of the vessel during evaporation.

Dr. W. R. Ormandy said that a considerable number of years ago he had had a great deal to do with the installation of water-softening plants, and the experience which he had thus gained made him very cautious in expressing any opinion upon the reactions which took place. Particularly was this true regarding the mutual reactions taking place between oxygen and carbon dioxide in the presence of iron. Unlike Professor Brame, he thought that hydrolysis played an important part. A previous speaker stated that he did not find much evidence of electrolytic effects. It seemed to him that the extraordinary effects of local corrosion, spotting, in a boiler must to some extent be attributable to the heterogeneous nature of the metal which was employed. He thought most of the members who had had a good deal to do with boilers would remember occasions when an old boiler, made in the days when puddle iron was used, resisted all the waters used in it and gave no signs of corrosion, but when that boiler was displaced by a steel boiler then entirely different results were obtained, even with the same water which had been used successfully in the wrought-iron boiler. It seemed to him that that might be attributed to the fact that the steel almost invariably contained segregations rich in manganese. It was known, not only from the experiments which had been described in the paper, but from other work that had been done on the subject, that manganese was an extraordinary material for acting as a carrier of oxygen. It was mixed with potassium chlorate in order to get oxygen off, and manganese sulphate was added in many reactions because it acted as an oxygen carrier. In many ways it was an ideal substance for the purpose.

It was difficult to grasp the details of the author's continuous process, but it was clearly intended primarily for power-stations and marine work where surface condensers were employed. He would like to point out, however, that more boilers were probably employed in producing steam for industrial operations where the condensed water could not be returned to the boiler. This led one to inquire as to the possibilities of the application of the de-oxygenising process in its application to such boilers. It was a very fortunate thing that the curve for the rate of the degassing reaction was such that at a temperature of 90° to 100° F., a very considerable velocity had already been acquired, because that was about the temperature at which every engineer desired the water to go into his economisers. It might, therefore, be assumed that water could be heated by waste steam to 90° F., sent through a de-oxidising machine, and then passed through the economisers. Incidentally, eighteen months or two years ago, when getting quotations for economisers, he came to the conclusion that the makers were going to get over the difficulty by silver-plating them inside.

He would like to know whether the author could say anything about the influence of traces of alkali on the rate of the removal of oxygen from boiler-feed water, because, as the members were aware, it was almost impossible to soften a water by any of the ordinary types of lime soda softening plants to within anything below 7 grains per 100,000 without having a certain amount of free alkalinity. He had met with a case of natural water which was so pure that it contained only 2 parts of solids per 100,000, but which seriously corroded steel boilers, and yet the addition of 2 grains per 100,000 of caustic soda inhibited that corrosion absolutely. He would like to know if the presence of free alkali in any way had that effect of stopping the absorption of oxygen in the degasser, or whether it necessitated the use of much larger degassers.

Mr. William H. Patchell (Vice-President) said that some of the members had been striving for a long time to obtain closer association between the engineer and the chemist, and that had been brought about on the present occasion. The *entente cordiale* had been much in evidence during the last five years, and it had led to surprising changes, and he was glad that through the good services of M. Kestner it had led to the Society of Chemical Industry joining with the Institution in the present meeting. Both Sir William Pope and Captain Sankey had said it was the inaugural meeting of the two bodies. He understood that it also set a precedent for the Society of Chemical Industry, in that he did not believe they had dared to meet anybody else before! He hoped the result of the combination would be thoroughly satisfactory.

M. Paul Kestner, in reply, said he was much obliged to the members for the kind reception that had been accorded to him. He was glad to hear the President, Sir William Pope, and Mr. Patchell agree with the suggestion he had made that the engineer and the chemist should work hand in hand, and that it would be of very great benefit to their industries. He would reply to the discussion in writing.

The meeting then terminated.

Communications.

Mr. R. W. Bailey wrote, in reply to Professor Brame's question relating to the character of the oxide coat, that he was struck by the remarkable uniformity and consistency in the action of their experimental deoxidiser, as shown by a period of operation of two to three months with only short stoppages at intervals for inspection or alteration in the number of sheets. During these runs no spottiness in the oxide coating occurred, which was very uniform in colour, red on the outside and greenish black below and next the metal. Probably the uniform conditions of flow, that could be obtained with expanded metal, accounted for the absence of a patchy coat. It would be interesting to know whether spottiness had been experienced with iron turnings, and he would like to ask for the author's experience on this point.

With respect to the physical method of degassing described by the author, namely, by exposing water to a reduced pressure, the action, of course, depended upon the magnitude of the partial pressure of the gas in the atmosphere to which the water under treatment was subjected, and this could be arranged to be very low indeed, although the total pressure might be appreciable. Thus water

exposed to an atmosphere of steam if finely divided would be degassed, because the partial pressure of gas there might be nil or very small. In appreciating the advantages of the author's continuous blow-down hot purification system of treatment, it was to be noticed that the inlet feed-water underwent degassing in this manner. To exploit this method to the full extent, however, it was necessary to draw off the gas liberated in order to have a low gas partial pressure. He would like to ask to what extent this was done. Instead of spraying water into an atmosphere of steam, a similar result, though not so complete, would occur if steam was bubbled through water. This happened in steam-boilers. A bubble of steam formed, having initially a gas partial pressure nil or very small, and as it rose it had a degassing action upon the water at its boundary. This was going on throughout the whole mass of the water, and thus it was that a large proportion of the dissolved gases was conveyed to the steam space and away to carry on corrosion of the plant beyond. This action of steam bubbles was not generally appreciated.

Messrs. J. H. Coste and W. E. F. Powney wrote that the general question of gases dissolved in water was discussed at length by one of the writers in 1917 and 1918.* Since then they had had occasion to investigate the nature and amount of both bases and solids dissolved in the water and associated with the steam in a large power plant. They were generally in agreement with M. Kestner, but on some points they were unable to confirm his views. The greatly increased velocity of the softening process when it was carried out at an elevated temperature was undoubted. They could not, however, think that many boiler waters contained so much calcium sulphate that it would be partially precipitated during the softening process. The solubility reached a maximum at about 40° C., and at 100° was about 120 per 100,000 of water.

The view that magnesium chloride was hydrolytically dissociated in dilute solutions with actual liberation of hydrochloric acid was, in their opinion, based on a misreading of Casaseca's Paper.† $MgCl_26aq$ seemed to be the stable form to 116·67° C. and between this and 181·5° $MgCl_2.4aq.$, after his $MgCl_22aq.$ See also Cribb (Analyst, xxv, 1900, 169). As properly softened boiler waters were usually more alkaline than $P_H=8$, the question of free acid could hardly come in. They were unable to agree with the view that the author appeared to hold that sodium carbonate alone was better for softening than a mixture of this and lime. Lime had the great advantage of adding no soluble salts to the water, of removing magnesia from solution, owing to the low solubility of magnesium hydroxide and of directly reducing the admittedly very small hydrion concentration due to carbonic acid. The corrosion of boiler plates or of turbine blades was undoubtedly due, as the author stated, to dissolved oxygen. Its action, however, appeared frequently to be that of a depolariser, removing the ferrous ion from solution and forming finally that oxide which was in equilibrium with water at high temperatures, Fe_3O_4. It was the presence of dissolved oxygen alone which allowed the action of carbonic acid or of saline electrolytes to be continuous.

They had found that water condensed in the very diminished pressure obtained in the condenser of a large turbine fitted with separate and efficient water- and air-pumps was almost free from oxygen, whilst that from a condenser fitted with a single pump for gas and water might even be super-saturated. The almost air-free water took up a considerable proportion of oxygen—about 25 per cent. of that required for saturation in flowing over a V notch and cascading down into the pipe leading to the feed-water heater. No doubt the turbulent flow was accountable for most of this, not the steady flow with unbroken surface over the V notch. In feed-water heater and open hot-well the concentration of dissolved oxygen would be about 70 per cent. of saturation. Some reduction, principally shown indirectly by rusting in the water, but also by the reduced proportion of oxygen relative to "nitrogen,"* was found at the inlet of the economiser. The water at the outlet was difficult to sample satisfactorily, but the steam delivered to a turbine had been examined and found to contain much less oxygen than would correspond to the nitrogen in solution, showing a loss of oxygen in the economisers and boilers. Some carbon dioxide was also found.

The writers' experience with condensers fitted with efficient air-pumps convinced them that, at least as a counsel of perfection, the soundest way of securing water free from dissolved oxygen was to allow no opportunity for the water, which could be condensed practically gas-free, to absorb any. That is, to use as far as possible a closed circuit. If the make-up water was kept down to a minimum, suitable chemical or physical means, which might be such as the author suggested, could be used for degassing it.

M. Kestner appeared to state that water at 100° C. and atmospheric pressure still retained a considerable proportion of oxygen in solution. This was correct in so far as the evolution of air required time, but there were several boiling-out processes for determining the gases dissolved in water which gave substantially exact results, although Leduc had shown that freezing was the most efficient means of degassing water. Of course, water at 100° C. under air, when the partial pressure of the air was 760 mm., would dissolve about 3 cm^3 per litre of oxygen. In any physical degassing process based on the use of an elevated temperature or reduced pressure or both, it was important to use appropriate means—spraying, dripping through small apertures, agitation, etc.—to obtain as large a surface of contact between the water and the atmosphere to which it was exposed.

Mr. Percy G. Jackson wrote that he did not entirely agree with the author's remarks in connexion with the continuous blowing-down of boilers. He had known cases in which this had been particularly useful, but he did not look upon it as generally necessary. The author stated that, in order that periodic blowing-off from the lowest point in the boiler might be effective, a large quantity of water must be blown off at each operation. In the writer's opinion this was not at all necessary. If the periodical blowing-off was for the purpose of preventing the accumulation of mud, the opening of the blow-off cock for a few seconds at a time was all that was necessary.

He deprecated most strongly the idea of returning the blown-off water to the boiler. Though the author removed the suspended impurities by passing the blow-off water through a settling chamber, he (the writer) did not see how any of the saline impurities could be removed, as the author appeared to suggest. No boiler should be worked with a saline concentration exceeding about 5000 grains per gallon, and as the solubility of all salts likely to be present in the water exceeded this, even at a temperature as low as 25° C., he could not understand how they could be removed by the simple process of cooling the blow-off water; and in the case of calcium sulphate, if present, this being more soluble at the lower temperatures than at the temperature of the boiler, it would have a tendency to re-dissolve rather than to be precipitated. As the prevention of undue accumulation of saline constituents was one of the two purposes for which blowing down

* Jour. Soc. Chem. Ind. xxxvi, p. 846, xxxvii, p. 170.
† Comp. Rend, 1853, xxxvii, p. 350.

* *i.e.*, nitrogen plus argon.

was desirable, it appeared that the object was defeated by returning it to the boiler. The lower the saline concentration in the boiler the better, particularly if nitrate were present. The heat lost by allowing blown-off water to run to waste was so slight, providing the blowing off was not excessive, that he could see no useful purpose in returning it to the boiler.

Mr. Philip L. Rivierf wrote that he would like to support very strongly all that M. Kestner had said on the necessity for closer co-operation between the chemist and the engineer. At the London County Council Tramways Power Station at Greenwich, where for some considerable time now they had been fortunate in having the assistance of a fully qualified chemist working under the supervision of the London County Council Chief Chemist, the results following on the chemical work carried out had been most gratifying.

With regard to the question of blowing-down boilers, they had had at Greenwich the usual difficulties in deciding on the proper quantity of water to be blown out from time to time, and they were not assisted to any great extent by considering the procedure adopted elsewhere, as there appeared to be no standard test adopted for the determination of the degree of concentration of salts in boiler waters, and no agreed definite limit to the degree of concentration of solids allowable before blowing down was considered necessary. As the result of the work carried out by the chemist, it was decided that the chlorine content of the boiler water should be taken as a measure of the solids present in the water, and a figure of 400 parts of chlorine per 100,000 parts of boiler water was taken as representing the concentration allowable in ordinary circumstances. When boiler water was analysed and the chlorine content ascertained, the "alkalinity" was also taken, and for the conditions existing at Greenwich, it had been found that chlorine + alkalinity × 3 = the total solids present in parts per 100,000.

When taking samples of water from boilers for analysis, it had been found that great care should be taken to ascertain which position on the boiler was most likely to give samples that would be consistent and furnish reliable figures on which the general condition of the boiler water could be determined. Under the conditions prevailing at Greenwich, where Babcock and Wilcox boilers were set in pairs and were fed through feed check-valves on each drum supplied by means of a pipe controlled by a hand regulated feed-valve for each boiler, it was found that the concentration of solids in the outside drum was usually from 4 to 5 times as high as the concentration in the inside drum. The analysis of the feed-water at Greenwich gave the following general results over a period of three months: alkalinity 0·91, chlorine 0·71, hardness 0·73, while for the same period the corresponding figures for the make-up feed were: alkalinity 6·8, chlorine 2·5, hardness 4·2, all figures representing parts per 100,000. It had been found that, in the case of the Babcock and Wilcox boilers, which were emptied and refilled with ordinary feed-water once every six or seven weeks when cleaning was carried out, the concentration of solids never exceeded 200 parts of chlorine per 100,000, and therefore no blowing down was necessary beyond opening and closing the blow-down cocks once per week to ensure that they were in working order.

In the case of the Stirling boilers, the allowable concentration had been temporarily restricted to 50 parts of chlorine per 100,000, pending an alteration to these boilers, and it was found that this figure was reached in from three to five weeks, depending on the work done by the individual boilers. When the concentration reached the figure given, the boilers were blown down to the extent of about 250 gallons each night for four nights, when it was found that the chlorine content had been reduced from 50 to 30 parts per 100,000, after which the boiler would usually steam for about from 10 to 14 days before blowing down was again necessary. In consequence of the comparatively small amount of water blown down, the amount of make-up feed used was a small percentage of the total water evaporated. Under modern conditions, apart from slight condenser leakage, the make-up feed being the only way in which solids could be fed into the boilers, the importance of restricting the amount of make-up feed required was at once apparent. From the results obtained at Greenwich, where the make-up feed was from 3½ to 5 per cent., it would appear quite reasonable to expect that in a large modern power-station the percentage of make-up feed should not exceed from 3 to 4 per cent. of the total water evaporated. The water blown out of the boilers did not as a rule exceed about 3·5 per cent. of the make-up feed, but this did not include the complete changing of water when boilers were laid off for cleaning.

The water blown out of the boilers when under steam, together with the water emptied out when boilers were emptied for cleaning, represented about 7·5 per cent. of the make-up feed. In order to keep a close check on the amount of make-up feed used and the amount of water blown out of the boilers, they had a meter on the make-up feed main and a "Lea" Recorder on the blow-down pipe-line, the introduction of the latter having proved particularly effective in preventing waste of water, through stokers having to lower the water level in banked boilers the feed-checks on which were leaking, and through the tendency to blow out water when the water level in working boilers had become dangerously high owing to lack of attention.

From their experience at Greenwich, it was difficult to conceive of a set of conditions which would justify the installation of M. Kestner's continuous blow-down system, as it would appear that the limit of concentration of solids in the boiler water would have to be considerably higher than was usually considered desirable, before the action of separating out he described could take place at the temperatures mentioned. The amount of water to be blown out in the ordinary way in a well-managed boiler plant would be so small that it would be very difficult to control the quantity within reasonable limits, as considerable trouble would be expected with regulating valves which would have to be operated under very severe conditions. He would like to know whether the author had determined the degree to which he considered the various solids contained in boiler waters might be allowed to concentrate before it was necessary to blow down through his apparatus, and which method of determining the degree of concentration he had found most reliable under ordinary operating conditions.

With regard to M. Kestner's proposals regarding the degassing of feed-water, it would appear that, in order to keep down the quantity of deoxidising material used to a reasonable figure, it was necessary to arrange either for a closed circuit system or that deäeration should be carried out to a certain extent by physical means before the water passed into the Paris degasser. As a result of very careful observation and readings taken over a period of seven days, it was found that, at Greenwich with a load curve as shown in Fig. 20, they required a hot-well capacity of about 1200 cubic feet to provide for the variation in the quantity of water carried in the hot-wells as reserve at different periods of the day. This hot-well capacity represented about 0·026 cubic feet per kw. of plant installed and appeared to be necessary, principally on account of the varying water-level in the boilers,

of which the feed was hand-regulated. Although no doubt the capacity required in the hot-wells could be reduced considerably by the introduction of feed-water regulators, they would still require to have a large quantity of water in reserve to provide for variation in demand on the hot-wells, and it was at this point that the difficulties of degassing appeared.

When condensate left the condenser it was, on account of the high vacuum now usually attained, almost quite free from oxygen, and in this state it was particularly liable to absorb a further quantity when exposed to the atmosphere, even for short

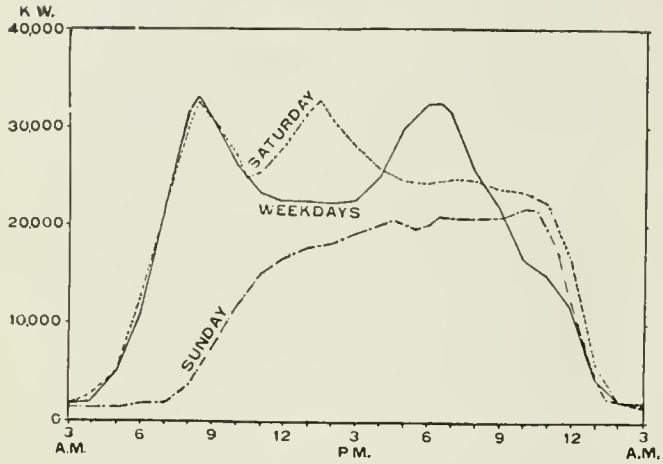


FIG. 20.

periods. Some time ago tests were made at Greenwich to determine the amount of oxygen present in the condensate from one of the turbine sets, and the following figures were obtained from samples taken at the points given:—

Condensate at—	Temperature of water ° F.	Dissolved oxygen. Cm³ per litre.	Lb. per million gallons.	Saturation Per cent.
(1) Lea Recorder Inlet	70	0·136	1·9	1·6
(2) Lea Recorder Outlet	70	2·14	3·0	26·2
(3) Feed-heater Outlet	133	3·82	54·6	91·0
(4) Hot-well.. ..	110	4·22	60·3	93·3

In taking tests to determine the amount of oxygen present in condensate from various sets, it had been found that, in the case of Edwards' air-pumps, dealing both with air and water, the condensate was frequently found to be super-saturated with oxygen, while in the case of sets where the air and condensates were dealt with by separate pumps, the amount of oxygen present in the condensate was almost negligible. The difficulty of storing water free from air, and at the same time allowing for considerable variation in level, was readily apparent, and the writer would be glad if M. Kestner could indicate in what way he proposed to provide for suitable hot-well capacity, such as was required in large power-stations.

M. PAUL KESTNER wrote that modern research in the metallurgy of iron and steel had been chiefly directed to improve their physical and mechanical resistance, and it was noticeable that this had been at the expense of their chemical resistance. Steel, which replaced wrought-iron everywhere today, had a much lower resistance to chemical reagents and the atmosphere than the latter, and there were wrought-iron fire-bars forged in the eighteenth century which were still in a perfect state of preservation. There could be seen, in sugar factories in the Colonies, rails and rolling stock made at the beginning of the sugar industry which were still doing good service, whilst similar material of recent origin had rusted after a very few years' use.

Referring to Professor Hinchley's remarks, the material used in degassers was not manganese cast-iron but manganese steel, cast-iron not being readily oxidisable when exposed to the air. The presence of graphitic carbon in general indicated a feebly oxidisable substance, even in the presence of manganese. Manganese steel in which the carbon was in the combined state was most favourable for oxidation. In addition, it would be impossible to obtain very long and thin turnings if manganese cast-iron were used. There were no traces of sulphur in the turnings used.

Replying to Mr. Arthur Spyer, good results could not be obtained if ordinary turnings were employed, as they tended to pack and channels were formed. Special turnings were therefore used made in the form of ribbon from 4 feet to 5 feet long. These gave a very homogeneous mass and had proved quite successful in practice. By the old process it was found that the turnings were used up and collected in heaps; the fillings also broke and crumbled on becoming rusty. Washing broke them still further, and the rust made them less able to absorb the oxygen. In the process of regeneration this was avoided; the mass kept its reducing properties sensibly constant until completely used up.

The distilled water produced in evaporators had the same oxygen content as the water before distillation, owing to the oxygen remaining in the vapour. This must therefore be degassed within the same limits as other boiler feed-water. If it were desired to avoid concentration in the boilers, the blow-down must be taken into account, particularly where boilers were using purified water only, without return of condensed water, as the boiler feed-water, after its passage through the purifier, contained soluble salts. These consisted of soda, sulphate of soda, and chloride of sodium. In general carbonate of soda was present in the greatest quantity; sulphate of soda only existed to any amount in water of great permanent hardness, and the chloride of sodium only existed in water which contained it originally, or which contained chloride of magnesium. In all purified waters there was carbonate of lime in solution, concentrated to saturation point at the temperature at which purification took place. All these salts concentrated in the boiler except the carbonate of soda, which concentration was limited by the continuous blow-down, and the carbonate of calcium, whose solubility was low and could not exceed its saturation at the boiler temperature. The latter salt gave rise to the formation of muds which were removed by the continuous blow-down and deposited in the purifier; thus there remained sulphate of soda and chloride of sodium. These salts concentrated until they were deposited in the purifier, due to the difference of solubility at the different temperatures obtaining in the boiler and purifier, these deposits being in proportion to the difference of saturation percentages at the two temperatures. This concentration represented for each of these two salts more than 300 grammes per litre. Such an increase would produce mud in the boilers and an elevation of the boiling point; thus it must be avoided.

Mud was produced in the boilers from the moment that a certain limit of viscosity was passed; and the latter was a function of the concentration and the temperature. The elevation of boiling point followed the Raoult's law. If one exterpolated for the saturation concentrations, and applied the formula

$$T = \frac{520 \times C}{M}$$

C being the concentration, and M the molecular weight of salt dissolved, then it was found for a

concentration of 1 per cent. an increase of 0·088 and for 30 per cent. 2·64. The elevation of boiling point corresponded to a very slight diminution of vapour tension and did not sensibly affect evaporation in the boilers. In practice it was found impossible to obtain perfectly dry steam, and, if the concentration in the boiler were raised, the water entrained by the steam would contain a larger quantity of dissolved salts and thus a larger quantity in the condensed water. The presence of these salts in the steam was apt to deposit sediment in the tubes of the superheaters and to attack the turbine blading. The concentration in the boilers must therefore be limited, the maximum depending on the initial concentration of dissolved salts in the purified water, and this limit was again dependent upon the type of boiler.

It has just been shown that there are numerous physical reasons for avoiding concentration in boilers; but on the other hand, reasons of a chemical nature opposed too low concentrations. The work of Friend and Brown and Heyn and Bauer had proved that there existed for each salt critical low concentrations for which the attack of the iron was a maximum, and that this attack diminished sensibly for higher concentrations. For carbonate of soda a concentration was rapidly reached, not only where the attack of the iron was stopped, but even where the soda protected the metal against the action of other salts; whilst the action of chloride and sulphate of soda decreased proportionately with the concentration, but at saturation point it was still appreciable. It was necessary, according to the above-mentioned authors, that the soda should reach a concentration in the boiler of more than 1 per cent., and it could be affirmed that for concentration of 1 per cent. the corrosive action of the other salts was sensibly lessened. This latter case corresponded with that obtained with continuous blow-down. To sum up, in the case of purification by continuous blow-down, the limitation of concentration in the boilers ought to be governed by physical reasons and be determined quantitatively for each case.

There were two methods, chemical and physical, for limiting the salt content in boilers:—

The *Chemical* method applied particularly to waters rich in sulphate of calcium. It consisted of introducing in the purifier carbonate of baryta which, by double decomposition with the sulphate of calcium, gave carbonate of calcium and sulphate of baryta. The two latter salts were extremely insoluble, and allowed complete elimination of the sulphate of calcium. No method was at present known for the elimination of chloride of sodium.

The *Physical* method was the simple one of blowing down the boilers periodically. The concentration in the boilers obviously would not increase if the quantity of salt which entered in a day was equal to that which was blown down in the same time.

Let N be the number of cubic metres of raw water introduced in the boiler in twenty-four hours.

c the concentration of the raw water in grammes of salts per cubic metre.

V the volume of the boiler in cubic metres.

C the concentration limit of the salts in the boiler in grammes per cubic metre.

$\frac{1}{p}$ the fraction of the volume of the boiler emptied by the purge in twenty-four hours.

The quantity of salts entering the boiler for twenty-four hours is

$$Nc.$$

The quantity of salts eliminated in twenty-four hours is

$$VC \times \frac{1}{p}.$$

Let these quantities be equated, then

$$Nc = CV\frac{1}{p}.$$

The concentration C found in the boiler at equilibriums will be

$$C = \frac{Nc\,p}{V}.$$

Note that $\frac{N}{V}$ is the number of boilers filled per day with the raw water

Let $$A = \frac{N}{V}.$$

then $$C = c \times A \times p.$$

The final concentration of the boiler would then be equal to the product of the concentration of the raw water by the number of boilers of raw water evaporated per twenty-four hours and by the number of blow-downs necessary to empty the contents of one boiler. In other words, it is proportionate to the concentration of the raw water by the output of the boiler, and inversely to the quantity of the blow-down.

The preceding calculation enabled the following curve to be drawn, Fig. 21. It was thus seen that the concentrations might be limited without important losses of hot water, and blowing down was not so important a factor where the salt content in the water was low. The installations most unfavourable were those which evaporated purified water and which did not recover the condensed steam. In those cases where the condensed steam was reintroduced into the boiler, it was only necessary to blow down a small quantity. Boilers using distilled water similarly only blew down a very small percentage, since the distilled water contained a very small quantity of salts. It had been found that continuous blow-down was the least costly method of purification, as much on account of the price of the plant as of their running cost. Added to these advantages was the fact that no incrustations were formed in the boiler and that consequently cleaning was unnecessary. Galvanic action did not appear to play any large part in the corrosion of boilers.

In reply to Mr. R. W. Bailey the Author stated that the employment of the special ribbon gave excellent results, and did not allow of short circuits or channelling (*see* reply to Mr. Spyer). The average relationship established between the speed of degassing at low and high temperatures was shown on Fig 13. So many factors intervened that it was impossible to obtain a general formula, and relationships could only be established for particular cases. Tests had been made on expanded metal, but the net cost was about three times that of iron turnings for the same output, and for the same weight the turnings had a surface about four times greater.

With reference to Professor Donnan's remarks, chemical degassing was inapplicable to drinking water; the corrosion in town's water pipes must be overcome, either by the action of an incrusting coating by a lining of cement in the iron piping, or by using armoured cement feeders.

In reply to Professor Brame, the Author stated that what took place in a boiler at 200° C. could not be observed; but when it was opened, rust was found as a result of the attack of the iron. It was probable that others had ascertained the transformation of the oxides of iron. Without referring to Gmelin or to Hermes, references would be found in the most recent works and more on a level with actual experience (*see* Bibliography, page 360—article of Von Siegmon). All possible products between FeO and Fe_2O_3 and their hydrates were produced in this blackish-green oxide. The product that had been examined had a percentage of

oxygen which did not correspond either to one of the oxides or one of the hydrates. In other cases, different results had been obtained. It seemed that the temperature played a great part in the degree of oxidation of the final product. At the present moment researches had not been finished on the subject of this state of oxidation of the iron.

With regard to the subject of chloride of magnesium, it had not been found that a small amount

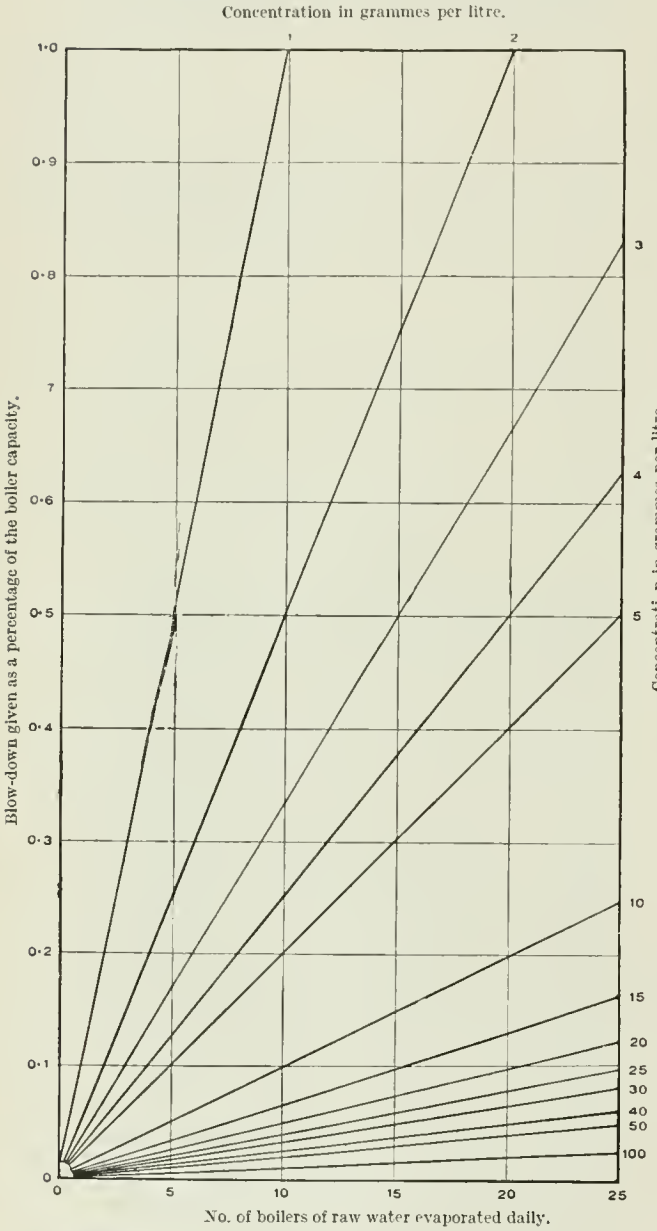


FIG. 21.
Concentration limit of water from boilers for the water circulation containing 0·1 gramme per litre, of dissolved salt.

of soda in the water modified the action. It was thought this was due to the very great oxidisable properties of the iron turnings.

In reply to Mr. Bailey's communication, irregularity or "spottiness" had never been observed in the layer of oxide in degassers where iron turnings were used; it always had a very uniform colour. Degassing by atomising the water in a space containing steam could only give good results by aspirating with a pump the gases and steam which were in contact with the water. This was done in the Elliott process by the use of a high vacuum and vaporisation of the water which had, under this small pressure, an almost explosive transformation into steam. The extraction of the steam necessitated a powerful pump; the condensed steam contained air which had to be eliminated. If it were sent again through the plant, a continual enrichment in air was produced in the water to be degassed. There was no doubt that good results had been obtained by this process, but it was open to the following objections:—

(1) It was difficult to obtain sufficient vacuum when the amount of water to be degassed was large.

(2) In this plant, where the water and the steam were under a very reduced pressure, particularly so in the water extraction pump, a very small leak sufficed to destroy the effect of the degassing.

In regard to the communication by Messrs. Coste and Powney, the facts quoted on the subject of sulphate of calcium were known; that was why in the text it was stated that the diminution of solubility of the $CaSO_4$ produced by the elevation of temperature was of very little importance in that case. The reference quoted on the subject of $MgCl_2$ was not known, but there had been no cases observed of corrosion by chloride of magnesium where the water was alkaline; but there were numerous examples in the case of collieries where the water was neutral, or slightly acid. On the other hand, it was not contended that purification by soda alone was better than by lime and soda; the point being that, in the particular case of continuous blow-down, purification by soda was more advantageous.

As to the condensed water regaining oxygen, it was very difficult to avoid in an ordinary or even specially made circuit; pumps and hot-wells of variable level were also causes of air re-entering. The question was solved in the following way:—

(1) Maintain a small pressure in the total water circuit; thus leaks could only take place from the interior to the exterior. All pumps should be under load as much as possible; thus there could be no re-entering of air.

(2) The degasser was under load and placed immediately in front of the feed-pump.

(3) For hot-wells of variable level in the circuit type there was always feed-water at high temperature; thus a cushion of steam could be maintained over this water to create pressure in the space of hot-well not filled by water and to avoid re-entering of air. The special hot-wells were combined with a degassing device placed therein, which removed the oxygen from the water before it left the hot-well; thus there could be large variations in the demand for water.

As for elimination of gases from the water by boiling, it must be remarked that this only took place completely by prolonged boiling, and that it would be impossible industrially to degass by such boiling, which would necessitate removal of an important quantity of water at the same time as the gas. An equilibrium was established between the partial pressure of the oxygen in the steam of the oxygen content of the water; in order that this should be nil, the steam must be pure. It must be noted that the oxygen content of the water was infinitely small in comparison with the volume of steam that this water represented.

Continuous blow-down was the cheapest process, and if it were a question of particularly difficult cases, as Mr. Jackson indicated, it was generally

advisable to apply it throughout. Continuous blow-down had the one aim of avoiding calcareous precipitation in the boilers. It was not a question of attaining concentrations, or sulphate of soda, for example, would be precipitated, but only of limiting the concentration of carbonate of soda and producing precipitation in the purifier by difference in temperature of part of the $CaCo_3$ of the blow-down.

With regard to Mr. Riviere's remarks, the question of purification ought not to be considered only from the point of view of central stations where 98 per cent. of condensed water was recovered, but from that of the more numerous works using purified water only, which did not recover their condensed water or use it for other purposes than feeding the boilers.

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ADDENDUM.

Since Mons. Kestner's paper was given in London on March 4, 1921, the Kestner system of boiler feed water purification and degassing has fully proved its worth in operation, often under exacting conditions. As an example, the installation at the 325,000 k.w. power station at Gennevilliers, near Paris, may be taken. At this station the complete Kestner system of purification, with continuous blow-down and degassing, has now been in operation since January, 1922, and the results obtained have fully justified its adoption.

The water supply is obtained from the Seine, and has an average hardness of 17°. After purification this is reduced to an average figure of 0·77. The type of purifier installed is illustrated in Fig. 1.

The results of tests taken of the dissolved oxygen present in the water showed that after passing through the Kestner degassers it was reduced to a figure which lay between one and two parts in ten thousand by volume.

The boilers are fitted with Kestner steel economisers, Dieterlen's patent. These are of an original design in which an extremely high rate of heat transfer and consequently low exit temperature of the flue gases are obtained. In order to examine the economiser tubes several of these were taken out and cut longitudinally, thus exposing to view the inner surface for a considerable length. This inspection showed that although the economiser had been in commission for a period of over twelve months not the slightest sign of corrosion was visible.

(1) Section of Kestner cylindrical type degasser.
(2) Degassers assembled in shops before despatch.
(3) and (4) Kestner water purifier for small outputs, 200—3000 gallons per hour.
(5) Outside view of same.
(6) Sectional view of cylindrical type purifier having a capacity of 13,500 gallons per hour.
(7) Outside view of same.

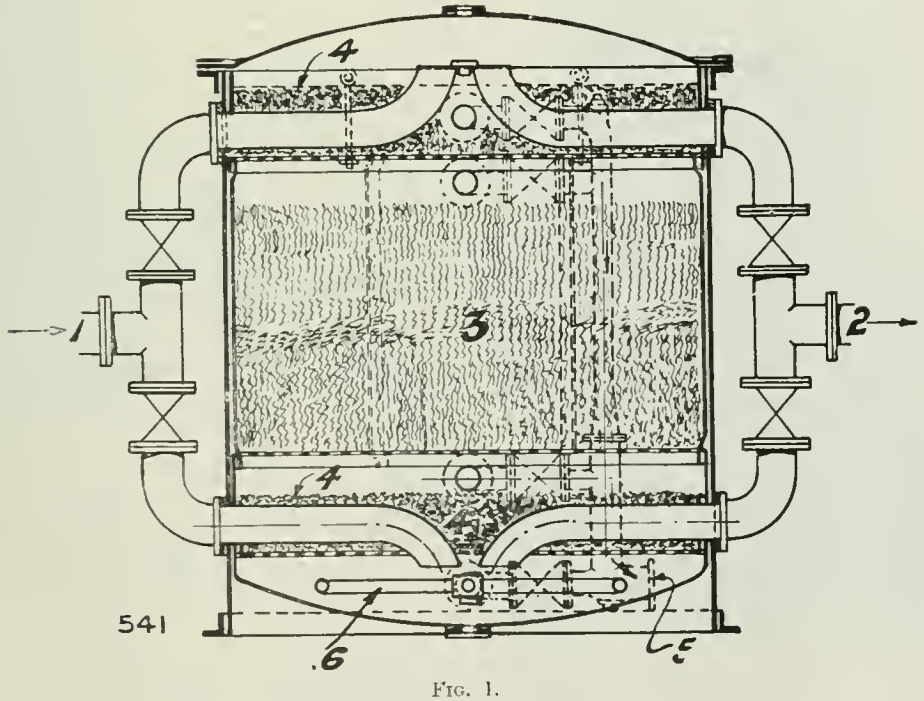


Fig. 1.

Section of Kestner cylindrical type degasser

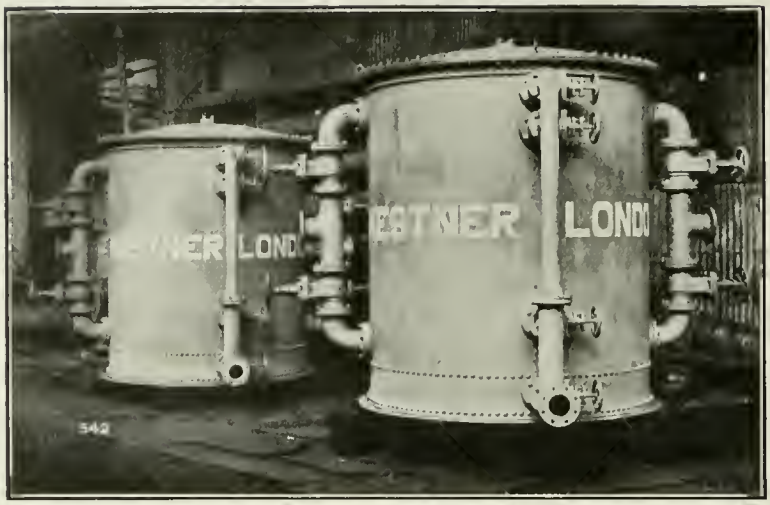
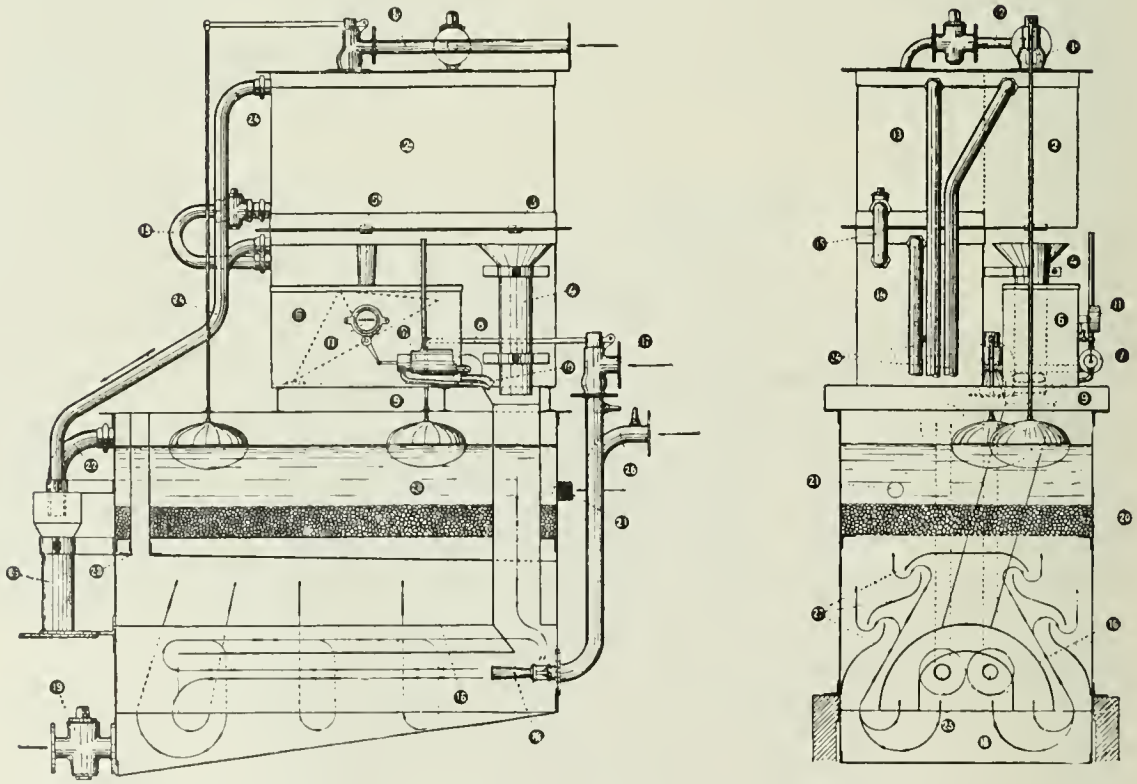


FIG. 2.
Degassers assembled in shops before despatch.



FIGS. 3 AND 4.
Kestner water purifier for small outputs, 200-3000 *gallons per hour.*

FIG. 5.
Outside view of same.

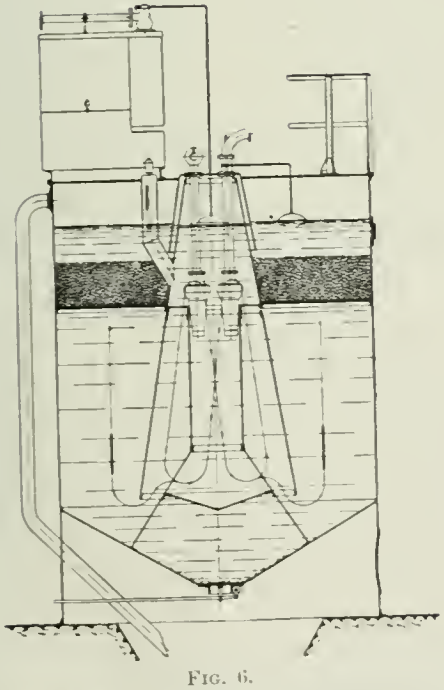


FIG. 6.
Sectional view of cylindrical type purifier, having a capacity of 13,500 gallons per hour.

FIG. 7.
Outside view of same.

1922

POTENTIAL DEVELOPMENTS IN REFRIGERATION.

By Dr. W. R. ORMANDY and E. C. CRAVEN.

(A paper read at a Joint Meeting of the Chemical Engineering Group with the Glasgow Section of the Society of Chemical Industry, at The Institution of Shipbuilders and Engineers, 39, Elmbank Crescent, Glasgow, on January 27th, 1922. Mr. J. Arthur Reavell, M.I.M.E., M.I.Chem.E., Chairman of the Chemical Engineering Group, in the Chair.

Although many means have been proposed for the commercial production of low temperatures, those now commercially employed are comparatively few in number. We can divide refrigeration machinery broadly into two classes—compression and absorption machines. The compression machines generally employ carbon dioxide, sulphur dioxide, ammonia or ethyl chloride, whereas the absorption machines are practically confined to the use of ammonia gas in conjunction with water.

The bulk of the refrigerating work of the world is carried out by carbon dioxide and/or ammonia compression plants. The carbon dioxide plant works at a very high pressure and is capable of producing very low temperatures, but the efficiency diminishes very rapidly with increase in the temperature of the condenser water. The ammonia compressor plant runs at a much lower average pressure than the carbon dioxide plant, and its efficiency is not reduced to the same extent by variation in the temperature of the condenser water. The carbon dioxide compression plant is not employed in naval work, since a war vessel might be sent at short notice, say, from the North Sea to the Red Sea, and the temperature of the circulating water in the Red Sea would make the use of the CO_2 plant practically impossible. Most navies refuse to employ the ammonia compression plant owing to the great danger of a breakdown accompanied by leakage of the ammonia gas into the confined spaces of the ship. It is, of course customary for the manufacturers to state that ammonia plants do not leak, but in practice leakages always occur some time or another, and generally, to a smaller degree, most of the time. Many navies use the ethyl chloride compression plant in spite of its somewhat lower efficiency and the larger size of plant necessarily employed. The lower pressure diminishes the chance of leakage and the gas which is set free, due to leakage, is not so obnoxious as is ammonia, but it has the drawback that the vapour and air form a combustible mixture.

The sulphur dioxide compression plant has largely gone out of use.

The absorption type of plant has hitherto made use of the rapid solubility of ammonia gas in water. The principle is simple, but to carry it out economically requires a somewhat complicated plant, and these complications are primarily brought about by the fact that on heating the watery solution of ammonia to drive off the ammonia gas, it is impossible to avoid driving off water vapour at the same time, and most of the complications are brought about in handling of the residual and distillate liquors.

A considerable number of years ago the facts that solid ammonium nitrate absorbs ammonia rapidly and is thereby converted into liquid compound was suggested as the possible basis of an absorption ice machine, and much money and effort was spent in endeavours to carry the idea into practice. It was found that commercially dry ammonium nitrate did absorb ammonia gas with great avidity and to the extent of something like 33% of its weight at ordinary temperatures. The liquid compound of ammonium nitrate and ammonia so produced, when heated to 100° C., gave off its ammonia against a back pressure sufficient to bring about liquefaction of the ammonia gas. As this reaction took place without any of the ammonium nitrate being decomposed or carried over with the gas, it appeared to have all the essential requisites for a satisfactory refrigeration process. Continual work, however, proved that the matter was not quite so simple as it appeared. There were conditions under which the amount of ammonia absorbed was insufficient to enable the process to be carried out commercially.

Before anything could be done towards the technical design of an adequate plant, the physio-chemical data in relation to ammonia and ammonium nitrate compounds were obviously requisite; the curves and data sheets which follow record some of the work done to obtain this necessary information.

Practical Considerations.

If ammonium nitrate is to be employed as the absorbent for ammonia in a technical plant, the most obvious course is to make the vessel containing the ammonium nitrate into an absorber and a generator alternately. An ammonium nitrate which is solid to commence with becomes liquid during the operation, and the ammonium nitrate crystals are heavier than the liquid double compound, the rate of absorption becomes very slow, for the ammonium nitrate crystals are protected by the liquid layer above them. To overcome this difficulty agitation of the contents is desirable, and the first commercial plants consisted of rotary cylinders having coils in the interior, through which steam or cooling water could be passed, according as the cylinder was to serve as a generator or absorber. In order that the process might be continuous, at least two, and preferably three, such vessels have to be employed.

Commercial plants working on these lines have been manufactured and have, on a two-ton plant, given 26 to 30 tons of ice per ton of coal employed.

It is very obvious that the use of rotary horizontal cylinders introduces undesirable complications, and efforts were made to carry out the process in a stationary plant containing no moving parts.

In the literature little is to be found regarding the compounds of ammonium nitrate and ammonia, and certainly not nearly enough to form a basis upon which to design a commercial unit. It was necessary to make a thorough investigation of the interaction between NH_4NO_3 and NH_3 both from the chemical and the physical standpoint.

As is well known, ammonia has the property of forming compounds with many salts by direct addition. With calcium chloride, for example, compounds containing 2, 4, and 8 molecules of NH_3 are formed. With aluminium chloride compounds with 1, 3, 5, 6, and 9 molecules have been reported. The compound with silver chloride will ever remain notable, since by the aid of this compound Faraday succeeded in liquefying ammonia in 1823. The equilibrium pressures for the two silver chloride compounds, $2AgCl.3NH_3$ and $AgCl.3NH_3$, have been determined by Isambert (Comptes rend., **66**, 1215, and **70**, 456), also by Hortsman (Ber., **9**, 749), and by other observers. It has been shown that for every temperature there is a certain pressure at which neither absorption nor evolution of ammonia occurs, but equilibrium is established. This equilibrium pressure is independent of the relative amounts of the two compounds present and is solely governed by the presence of the compound which contains most ammonia. The difference between the equilibrium pressures of the two compounds at any temperature is so great that it is not difficult to study the dissociation relations.

In some cases however experiment has shown that it is almost impossible to determine equilibrium pressures for various temperatures owing to the slowness and irregularity of changes in such pressures. The following figures selected from Weinhold's work on $CaCl_2.8NH_3$ (Pogg. Ann., 149, 220) illustrate this point:—

	Temp. °C.		Equilibrium pressure mm.
Temp. rising	0	..	121
	15·7	..	295
	17·8	..	320
	21·4	..	380
	43·4	..	1283
Temp. falling	21·5	..	599
	19·9	..	436
	16·3	..	310
	0	..	170

It cannot be doubted that the great differences between these figures show that equilibrium was not established, but was only apparent owing to the extreme slowness of the attainment of true equilibrium.

In approaching the subject therefore of the dissociation pressures of other ammonia addition compounds we would naturally expect to find a definite temperature-pressure relation for each compound formed, providing that the true equilibrium pressure was attained in reasonable time. The compound however on which we purposed working has this remarkable difference from the bodies already mentioned, that it is liquid at ordinary temperatures. The ammonia nitrate compound is not unique in this respect, ammonium iodide and bromide, sodium iodide, potassium sulpho cyanide, and other salts giving liquid compounds with ammonia.

When ammonia gas is passed over cooled ammonium nitrate, a colourless, rather mobile and highly refractive liquid is formed. At atmospheric pressure and ordinary temperature this liquid was found by M. Troost (Comptes rendus 94, pp. 789) to possess the composition $2NH_4.NO_3.3NH_3$. He determined the freezing point of the liquid as 22° C. and obtained the following equilibrium or vapour pressure figures:—

Temp. ° C.		Pressure. mm. Hg.	Temp. ° C.		Pressure. mm. Hg.
−30	..	90	10·1	..	525
−26	..	115	14	..	600
−18	..	170	18·4	..	715
−10	..	250	20·8	..	765
−0	..	365	25	..	930

It is also stated that a compound $NH_4.NO_3.3NH_3$ appears to be formed at lower temperatures.

M. Raoult (Comptes rend. 94, 1117) gives the following determinations of ammonia absorbed by ammonium nitrate at various temperatures, the pressure being 745 mm.:—

Temp. ° C.		Ammonia absorbed per 100 $NH_4.NO_3$.	Temp. ° C.		Ammonia absorbed per 100 $NH_4.NO_3$.
0	..	48·1	18	..	32·0
6	..	42·0	24	..	28·0
12	..	32·8	30	..	18·0

Kuriloff (J. Russ. Chem. Soc., 25, pp. 170—192) states that at 0° C. compounds or mixtures containing between 27 and 9 parts of ammonia per 100 parts ammonium nitrate, have the same vapour pressure.

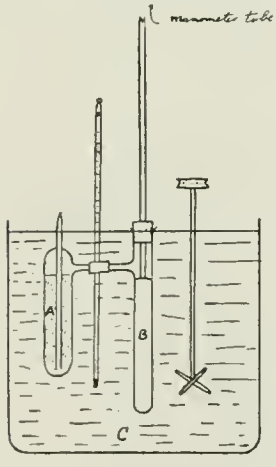
These few notes are the main facts to be found in the literature with reference to the present work.

Experimental.

The first experiments made in determining the vapour-pressure of the $NH_4.NO_3/NH_3$ liquid consisted in preparing the liquid in a light glass tube which was then introduced into a steel bomb. A pressure gauge of the Bourdon type was fitted. The bomb was heated in a water bath to various temperatures and the pressure noted. Various sources of error were found which gave rise to bad results. The errors arose from:—

1. Defects of gauge.
2. Partial pressure of air in bomb.
3. At high pressures, condensation of liquid NH_3 in gauge.

After further trials it was decided to make a series of pressure measurements not exceeding $2\frac{1}{2}$ atmospheres in a glass apparatus by means of a mercury column. The apparatus was arranged as shown in the sketch for low pressure measurements, Fig. 1. The nitrate was taken in the bulb A, and



Apparatus for low pressure measurements.
A.—Bulb with kieselguhr mixture.
B.—Mercury manometer connected by wired on pressure tubing.
C.—Bath with thermometer, etc.

FIG. 1.

dry NH_3 passed in by the sealed-in tube. When the liquid compound had formed fully, mercury was poured in through the tube of the manometer while

a stream of NH_3 was passing. This precaution obviates the possibility of any air being carried into the apparatus by the mercury. The NH_3 leading-in tube was then sealed. The bath was heated to various temperatures and the corresponding pressures read off from the height of the mercury column. With the fully liquid bodies equilibrium was observed to be attained in a very few minutes, and the pressure readings followed slight variations in temperature with very little lag. This shows that we had true equilibrium conditions established.

The following results were obtained:—

(1). ° C.	Height. Hg. mm.	Pressure. mm.
15	"small"	963
20	196	963
25	357	1124
30	548	1315
35	745	1522
40	970	1737
45	1230	1997
50	1520	2287
55	—	—

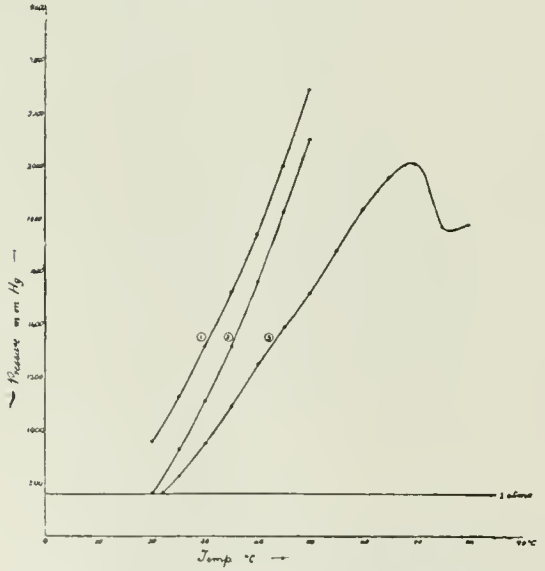
Some NH_3 was now driven off from the liquid by unsealing the tube. After resealing the following curve was taken:—

(2). ° C.	Height. Hg. mm.	Pressure. mm.
20	0	760
25	168	928
30	348	1108
35	554	1314
40	800	1560
45	1065	1825
50	1350	2110

This curve follows on well from that given by Troost. Further ammonia was let off till a considerable quantity of solid ammonium nitrate had separated. The following results were obtained:—

(3). ° C.	Height. Hg. mm.	Pressure. mm.
22	0	765
25	60	825
30	185	950
35	322	1087
40	480	1245
45	630	1395
50	750	1515
55	910	1675
60	1070	1835
65	1190	1955
70	1340	2005
75	1000	1765
80	1010	1775

These results are plotted on Graph 1.



Low pressure curves of liquid bodies without kieselguhr.
(1) About 38pts. NH_3 per 100 NH_4NO_3
(2) „ 30 „ „ „
(3) „ 12 „ „ „

GRAPH 1.

The following notes were made during the taking of curve (3), and may serve to throw a little light on the strange result:—

"Pressure increase rate falling off after 45° C. Surface of liquid clouds over. Pressure becomes uncertain. On shaking liquid pressure rises about 50 mm., the highest reading being taken in each case. Ammonium nitrate begins to float in liquid 55—60° C., and is obviously dissolving. At 60° C. a thick mush resulted. Pressure varying 15 cms. Above 65° C. an almost dry looking porous mass formed and pressure dropped."

It was further noted that on cooling down the pressures were much lower than at corresponding temperatures when heating up.

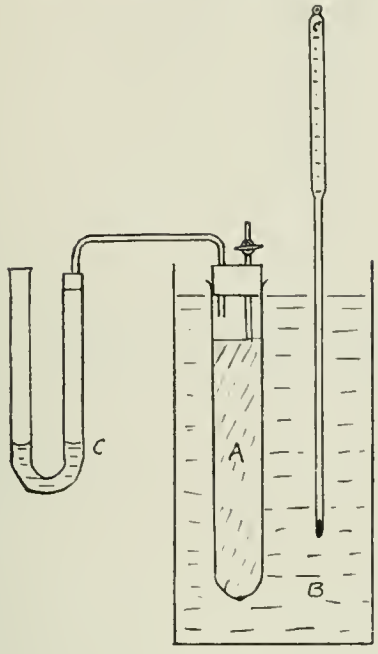
It seems impossible to avoid the conclusion that ammonium nitrate heated with a moderate amount of the liquid compound under certain conditions, may pass into a cellular state favouring the absorption of ammonia, so that the full vapour pressure of the liquid compound is not exerted. This change of state is possibly connected with the well-known transitions of crystalline state of ammonium nitrate and with the formation, as we shall show later, of a lower compound $3NH_4.NO_3.NH_3$. It was noted that the liquid $2NH_4.NO_3.3NH_3$ compound heated to 30° C. at atmospheric pressure leaves a dry porous residue containing about 5% of NH_3. This body on standing at ordinary temperature for 24 hrs. breaks down to a crystalline mass of ammonium nitrate wet with the 2:3 liquid. It is possible that in our experiment that at 65—70° C. and the pressure stated, a similar change occurred. Moreover, as we shall also show by further experiments, the porous mass of ammonium nitrate left after driving off ammonia from the 2:3 compound, absorbs ammonia several times as fast as the original crystals of nitrate.

It was obvious that to extend our pressure measurements over a wide range of compositions some method of preventing the formation of the porous variety of ammonium nitrate would have to be adopted. The method adopted was furnished by some unpublished work performed by one of us, and consists in absorbing the 2:3 liquid compound in kieselguhr in such proportions that a dry powder is formed. Owing to the extremely fine division thus ensuing, the possibility of the formation of nitrate masses, porous or otherwise, is reduced to a minimum.

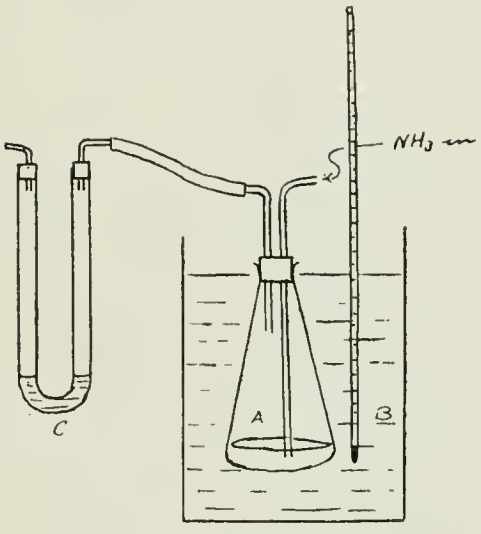
Some preliminary experiments showed that, using kieselguhr as employed for dynamite manufacture, suitable proportions are:—1 part kieselguhr, 2 parts 2:3 compound or 1·5 parts ammonium nitrate. In all subsequent experiments, the kieselguhr mixtures were made in this proportion. The most convenient method for the preparation of the absorptions was found to be as follows:—A weighed amount of ammonium nitrate dried at 120°—130° C. is mixed with the calculated amount of kieselguhr. Dry NH_3 is then passed through the mixture so that the liquid body is absorbed in situ as fast as it is formed.

Some of the mixture made in this way in an apparatus as shown in Fig. 2 was heated to various temperatures to determine the equilibrium composition under atmospheric pressure. The usual corrections were applied to the weighings. It was observed that true equilibrium was attained at each temperature in something under half an hour, the evolution of NH_3 ceasing and slight fluctuations of the glycerol in the U tube followed very closely small variations in the temperature of the bath. The following results were obtained:—

° C.	Parts NH_3 per 100 NH_4NO_3.	° C.	Parts NH_3 pe 100 NH_4NO_3
13	38·5	25	6·1
14	37·3	27	5·4
15	35·4	30	5·1
17	32·5	35	3·5
19	31·1	40	2·5
21	30·1	50	1·8
23	25·6	70	0·9



Apparatus for equilibrium comp., temp. at N.P.
A.—Tube with kieselguhr mixture.
B.—Bath and thermometer.
C.—U-tube with glycerol.



Apparatus for rate of absorption.
A.—Flask with nitrate or mixture.
B.—Bath.
C.—Glycerol U-tube.

FIG. 2.

The residue was extracted with water and titrated with standard acid and showed 0·91 parts NH_3 per 100 parts NH_4NO_3.

A repeat made on a somewhat smaller scale gave the following results at lower temperatures:—

°C.	Parts NH_3 per 100 NH_4NO_3.	°C.	Parts NH_3 per 100 NH_4NO_3.
0	50·8	13	38·2
4	48·7	20	30·7
7·5	42·6	30	6·7
10	40·6	40	2·7

A curve constructed from these figures will be found on Graph II. The rate of change of NH_3 content, plotted against temperature is also shown.

A study of these curves shows that definite breaks indicating new compounds occur around the following figures:—

°C.	Parts NH_3 per 100 NH_4NO_3.		
0 (?)	50·8	2:5 =	53
9	41·5	1:2 =	42·5
20	30·5	2:3 =	31·9
27	5·5	3:1 =	7·0

There can be little doubt that the compounds having the molecular ratios of nitrate and ammonia indicated at the side are formed. Considering within what a small range of temperature all are formed or decomposed it is probable that their molecular weights are much the same. This would lead us to suspect some such series as:—

$$6NH_4.NO_3.2NH_3,$$
$$6NH_4.NO_3.9NH_3,$$
$$6NH_4.NO_3.12NH_3,$$
$$6NH_4.NO_3.15NH_3,$$

and perhaps, according to Troost,

$$6NH_4.NO_3.18NH_3.$$

Reverting again to pressure measurements, the low pressure apparatus was charged with the nitrate-kieselguhr mixture. Ammonia was passed in as before and the tube sealed off. Pressure temperature readings were taken. After each set of readings was completed, some ammonia was allowed to escape and a new set made. Thirteen curves were taken in all, the figures being as follows:—

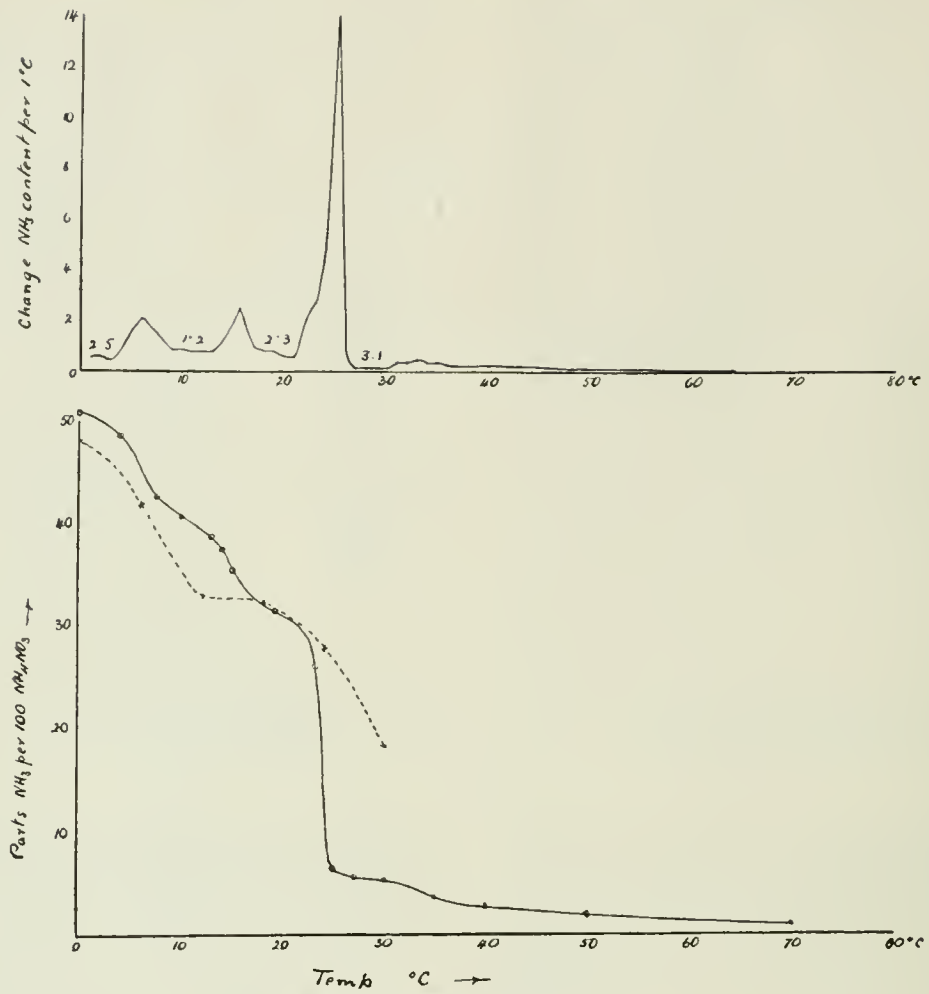
(1).		(2).	
°C.	Pressure. mm.	°C.	Pressure. mm.
0	755	0	—
6	1005	3	755
10	1145	10	1015
15	1350	15	1235
20	1600	20	1465
25	1890	25	1750
30	2200	30	2080

(3.)		(4).	
°C.	Pressure. mm.	°C.	Pressure. mm.
6	755	9·5	755
10	899	10·0	—
15	1071	15	925
20	1295	20	1105
25	1545	25	—
30	1820	30	1555
35	2095	35	1815
		40	2155

(5).		(6)a.	
13	760	14	798
20	952	20	966
25	1140	25	1147
30	1330	30	1348
35	1570	35	1584
40	1820	40	1840
45	2145	45	2164

Although set out as one series, these curves were really determined in two sets, 1—5 and 6a—13. 5 and 6a represent the meeting points of the two sets. As they are practically duplicate (6)a is omitted from the graphed curves.

(6).		(7).	
15	767	17	767
20	911	20	847
25	1087	25	1030
30	1296	30	1237
35	1528	35	1456
40	1792	40	1744
45	2100	45	2058



Variation of composition with temperature at N.P.
Broken curve—M. Raoult.

GRAPH II.

(8).			(9).		
°C.		Pressure. mm.	°C.		Pressure. mm.
21	..	767	24	..	767
25	..	884	25	..	797
30	..	1044	30	..	954
35	..	1243	40	..	1332
40	..	1482	50	..	1864
45	..	1755	55	..	2207
50	..	2064			

(10).			(11).		
25·3	..	767	28	..	767
30	..	898	30	..	837
40	..	1117	40	..	1037
50	..	1497	50	..	1351
60	..	2119	60	..	1748
			70	..	2307

(12).			(13).		
31	..	767	40	..	767
40	..	962	45	..	880
50	..	1259	50	..	1000
60	..	1593	60	..	1275
70	..	1966	65	..	1407
75	..	2213	70	..	1570
			75	..	1737
			80	..	1902

These curves have been plotted on Graph III, and show clearly the gradual variation of pressure with composition characteristic of the true compound, so different from the solid compounds considered at the beginning of this paper. This "damping" of the sudden change of dissociation pressure in passing from one compound to the next is no doubt due to the solubility of the ammonium nitrate in the liquid compound (6:9) and to the miscibility of the liquid compounds.

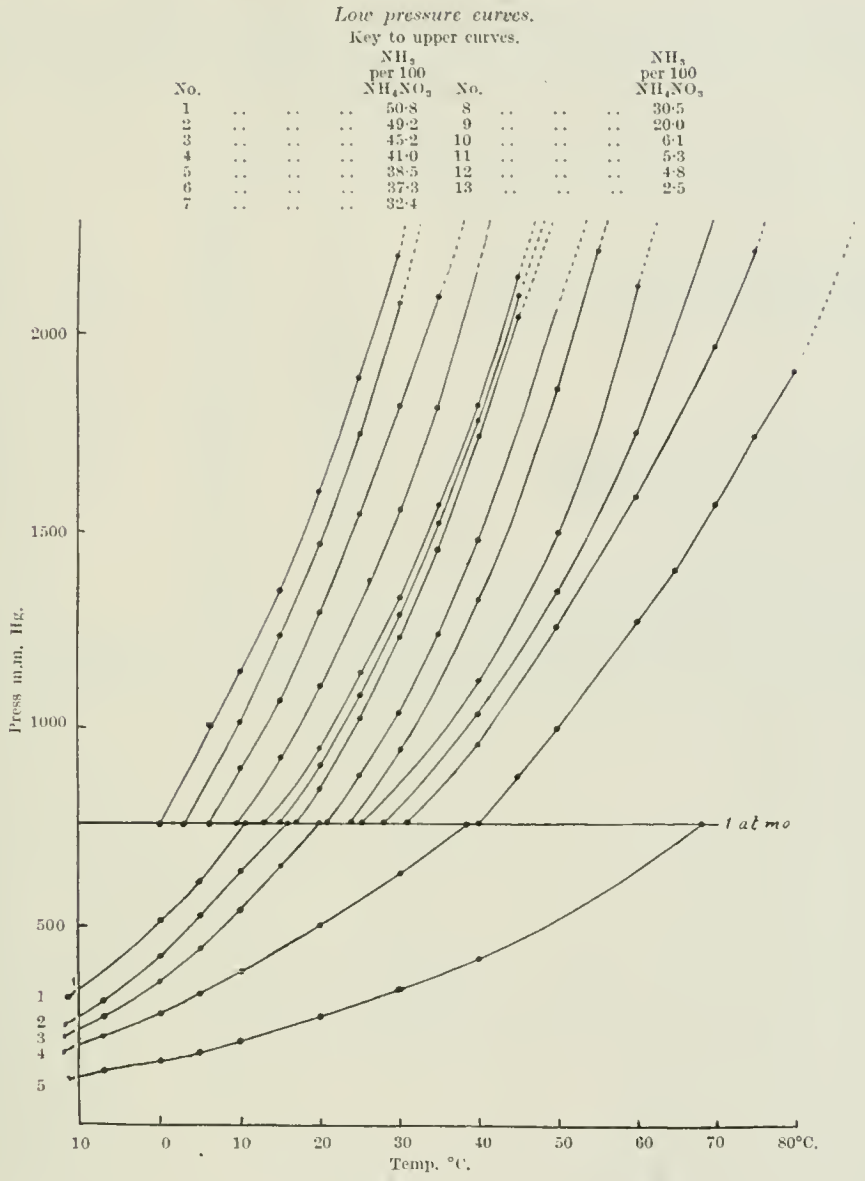
By a slight change in the apparatus, viz., the substitution of a sealed for the open manometer tube, we were enabled to follow the pressure curves below atmospheric pressure. The following results were obtained:—

(1).			(2).		
°C.		Pressure. mm. Hg.	°C.		Pressure. mm. Hg.
11	..	760	16	..	760
10	..	740	10	..	640
5	..	610	5	..	530
0	..	515	0	..	425
− 7	..	360	− 7	..	310
−12	..	320	−12	..	250

(3).			(4).		
19·5	..	760	38·5	..	760
15	..	650	30	..	635
10	..	540	20	..	505
5	..	445	10	..	390
0	..	360	5	..	333
− 7	..	270	0	..	280
12	..	220	− 7	..	220
			−12	..	180

(5).		
68	..	760
40	..	420
30	..	340
20	..	270
10	..	210
5	..	180
0	..	160
− 7	..	135
−12	..	110

These figures are also plotted on Graph III.



Graph III.

Considering the figures so far obtained with reference to the employment of ammonium nitrate as an absorbing medium for a refrigerating machine, we see that as regards the absorption end of a refrigeration cycle the temperatnre pressure relations include all conditions likely to be met in practice. On the other hand, for the evolution end of the cycle it is necessary to extend the measurement of vapour pressure to such pressures as are possessed by liquid ammonia at ordinary temperatures, say, 12 atmospheres.

Being unable to procure any accurate form of gauge capable of measuring these pressures, we fell back on the idea of measuring the compression of a column of air which affords a measure of the pressure by calculation from Boyle's law.

After some trials the apparatus took the form shown in Fig. 3. The steel bomb was filled with the ammonium nitrate—kieselguhr mixture, and ammonia passed through at a low temperature. The bomb was then immersed in a bath at 25° C. or so. A brisk evolution of NH_3 thus set in, during which evolution the capillary tube gauge was slipped into place. This precaution ensured that no air was left behind in the bomb.

A further advantage arising from the use of a gauge of small capacity is that the composition of the mixture must remain practically the same at high and low temperatures owing to the free space being small.

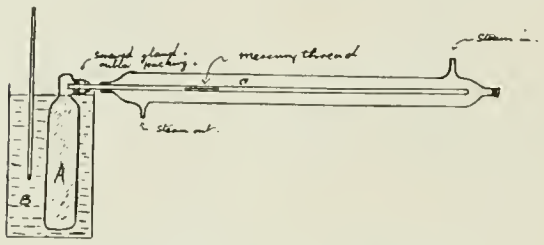
The capillary tube was furnished with a mercury thread some 5 cms. long to serve as an index, and

the whole tube was enclosed in a steam jacket as shown. This serves to prevent the condensation of liquid ammonia within the gauge, and also gives the following advantages:—

1. The air column is kept at constant temperature.

2. The air being heated more nearly obeys Boyle's Law.

The capillary tube was carefully calibrated over the entire length of the bore in the usual manner.



Apparatus for high pressure measurements.
A.—Steel bomb with kieselguhr-nitrate mixture.
B.—Bath with thermometer, etc.
C.—Capillary tube, steam jacketed.

FIG. 3.

The correction thus found for the length of the air space in no case exceeded 1% of the length involved.

The length of the air column in the pressure apparatus was measured by a simple form of horizontal cathetometer. As a rough check on the arrangement and calibration of the tube the expansion of the air column was measured between 15° C. and 100° C., and gave the value ·00360 per 1° C. (uncorrected). The expansion of the glass tube being unknown an exact figure cannot be calculated.

With the apparatus as described, the following figures were obtained:—

(1) 31·5% NH_3.		Pressure, mm. Hg.
° C.		
15	..	673
17	..	731
20	..	815
30	..	1170
40	..	1635
60	..	3120
80	..	5810
90	..	7420
95	..	8920

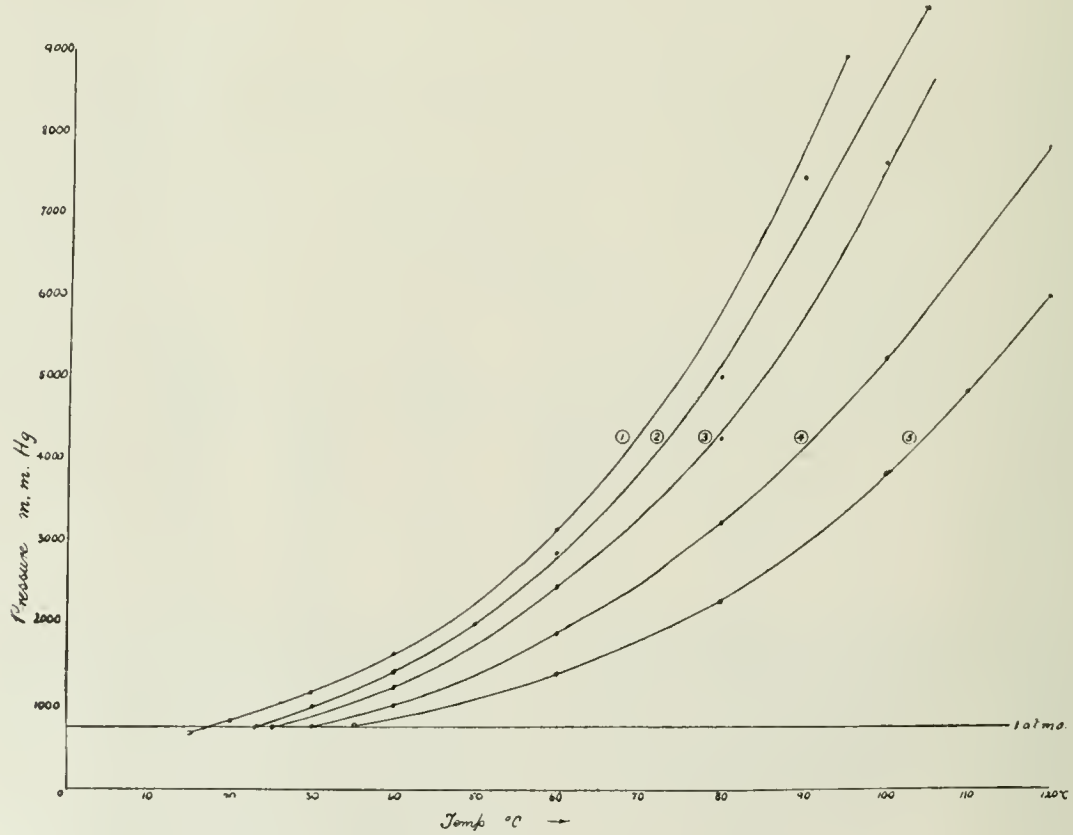
(2) 25·5% NH_3.		Pressure, mm. Hg.
° C.		
23	..	762
30	..	1015
40	..	1400
50	..	1975
60	..	2850
80	..	4980
100	..	9530

(3) 6·5% NH_3.		
25	..	762
40	..	1220
60	..	2430
80	..	4240
100	..	7620
105	..	8710

(4) 5·2% NH_3.		
30	..	766
40	..	1015
60	..	1875
80	..	3210
100	..	5230
120	..	7830

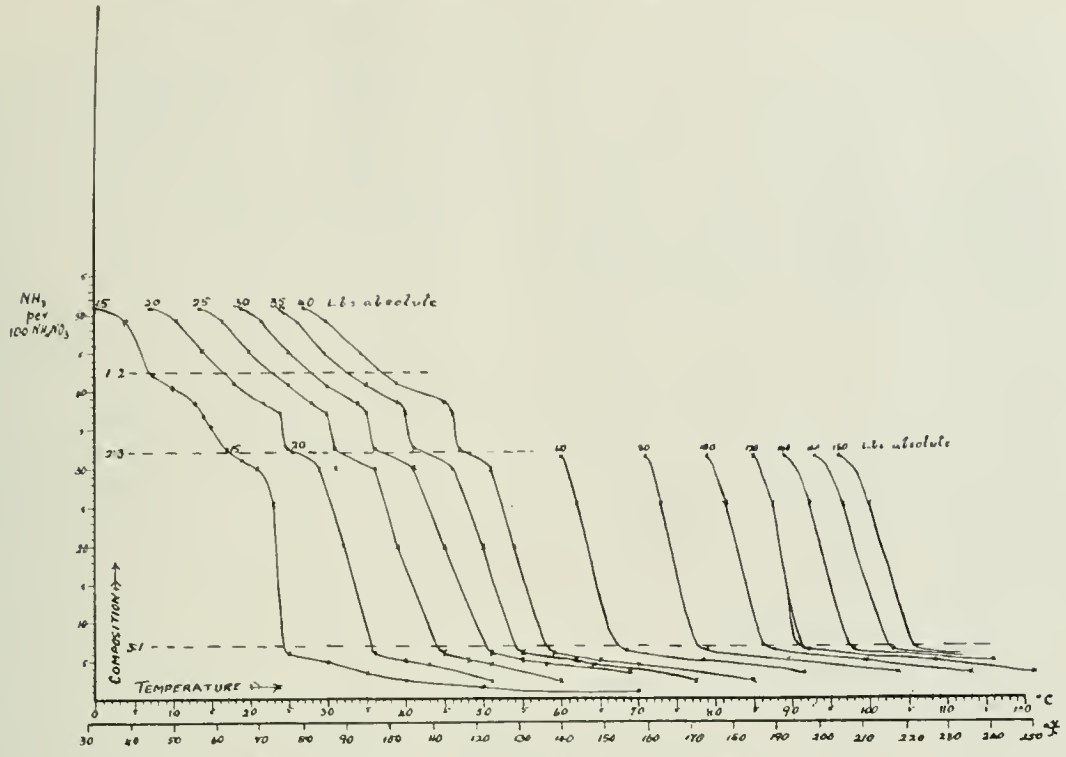
(5) 3½% NH_3.		
35	..	773
60	..	1400
80	..	2260
100	..	3830
110	..	4820
120	..	6010

These curves are plotted on the accompanying graph. (Graph IV.)



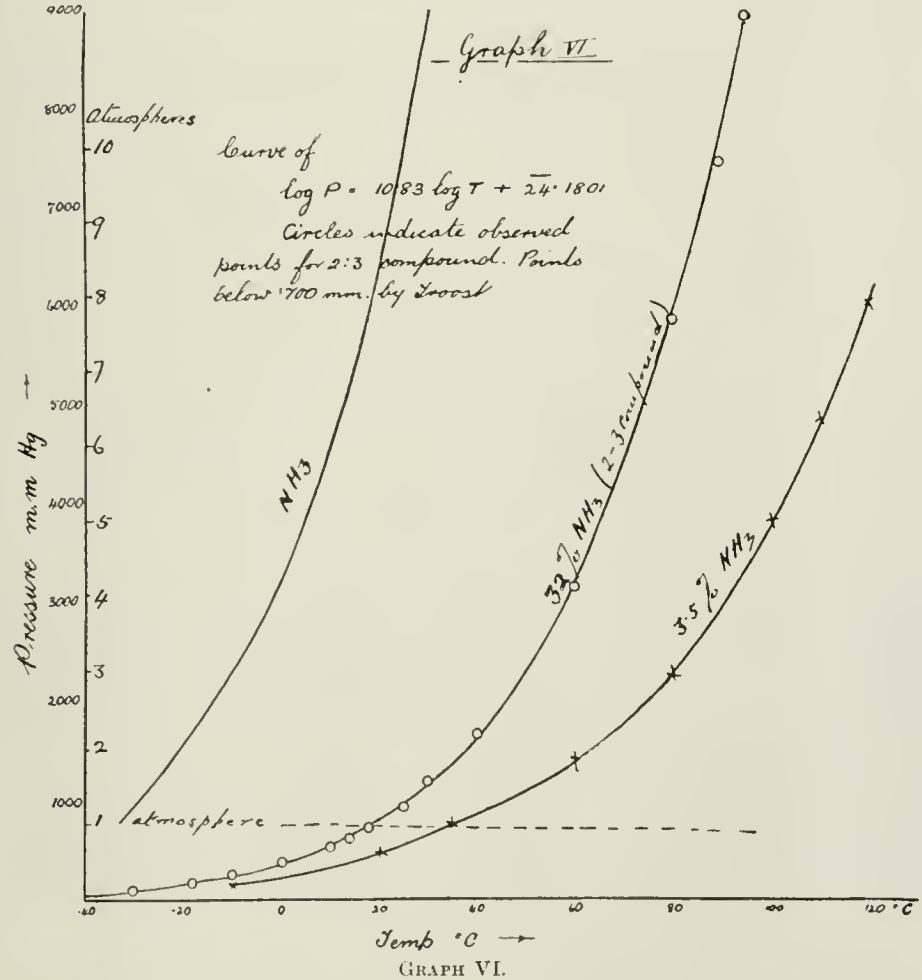
High pressure curves.
NH_3 per 100 Am. Nitrate.
(1)=31·5. (2)=25·5. (3)=6·5. (4)=5·2. (5)=3·5.

GRAPH IV.



Composition—Temperature Curves at Various Pressures (Absolute) in Kieselguhr.

GRAPH V.



GRAPH VI.

Considering both sets of pressure curves, and taking composition and temperature values as co-ordinates, we may plot isobars in the field, as shown in Graph V.

Isobars are plotted for ½, 1, 2, 4, and 8 atmospheres. It will be noticed that these are roughly parallel and equidistant, pressure thus increasing in geometrical progression, while temperature increases arithmetically, indicating a connecting equation of hyperbolic form.

The temperature pressure figures themselves are connected by an empirical expression of the form $\log P = a \log T + B$. The value of a remains constant over the range of liquid compounds, but falls off for lower NH_3 content than that possessed by the 3:1 compound.

In Graph VI. the curve of $\log P = 10{\cdot}83 \text{ Log } T - 23{\cdot}8199$ is plotted.

P is in mm. mercury and T in ° C. absolute.

The observed points for the 2:3 compound are marked, and show fair agreement.

Rate of absorption of NH_3:—

A few experiments have been performed in this direction. By attaching a manometer to the high pressure bomb during the period of charging with NH_3, we were able to observe in the following manner the variation of the rate of absorption with pressure. A moderate pressure of NH_3 having been set up the NH_3 supply was shut off. The rate of fall of the mercury column was then noted. The weight of nitrate present being very large in proportion to the weight of free NH_3 the composition of the absorbing mass could not have changed appreciably during this period. A study of the curves thus obtained showed that the rate of absorption varies directly with the pressure of NH_3 present. As this result might have been taken for granted it is unnecessary to elaborate this point further.

The result of passing NH_3 at a slight positive pressure over equal amounts of ammonium nitrate in various forms is shown on Graph VII. It will be noticed that the cellular form of nitrate absorbs much more rapidly than the crystalline.

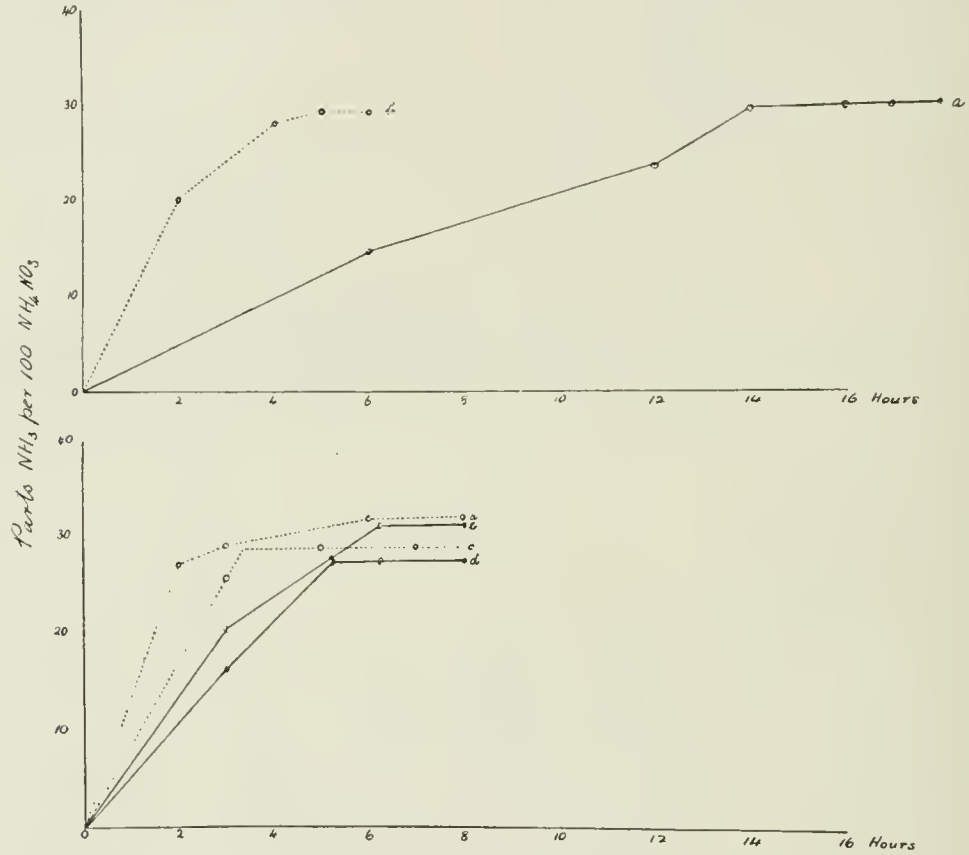
The specific gravity of the liquid compounds has been roughly determined. With 50 parts NH_3 per 100 $NH_3.NO_3$ the S.G. is 1·080 at 0° C., and with 28 parts NH_3 S.G. is 1·193 at 20° C.

The specific gravity at intermediate points follows the relation

$$S.G. = 1{\cdot}411 - P/100$$

where P = % of NH_3 in the mixture.

If a measured volume of the liquid bodies be weighed and the NH_3 then gently boiled off it will be observed that for each 1 c.c. diminution in volume a loss of about 0·7 gms. (695) of NH_3 will be sustained. This seems to indicate that the ammonia



Showing rate of absorption of NH_3.

a=NH_4 NO_3 crystals alone.
b=NH_4 NO_3 cellular form.

Kieselguhr mixtures.
$a+b$. 1 kghr. 1·5 NH_4 NO_3.
$c+d$. 1 kghr. 2·25 NH_4 NO_3.
Dotted curves. 2nd absorption.

GRAPH VII.

in the compounds occupies very approximately the same volume that it would have if liquid and somewhat below its b.p.

Below 28 parts NH_3 per 100 NH_4NO_3 the product is no longer entirely liquid at ordinary temperatures.

The liquid body having the sp. gr. 1·193 at 20° C. when cooled to 10° C. had a gravity of 1·209—a variation of ·0016 per ° C. Liquid NH_3 at the same temperature varies ·0014 per ° Centigrade.

Application.

Generation of NH_3.

Against back pressures up to 190 lb. per square inch absolute, *i.e.*, cooling water at 90° F., all the ammonia-ammonium nitrate bodies containing over 6% of ammonia are decomposed at a temperature of 230° F., *i.e.*, steam at 21 lb. per square inch absolute or 6 lb. pressure on the gauge. The heat required to drive off the remaining percentage grows very rapidly, and it would be economically unsound to attempt it.

With cooling water at 76° F. (144 lb. absolute back pressure) all the bodies over 6% NH_3 are decomposed below the boiling point of water.

In order to decompose the ammonium nitrate-ammonia compounds, especially those containing lower amounts of NH_3, it is necessary to agitate during the evolution period to avoid the formation of layers or to mix the compound with some absorbent such as kieselguhr. A stationary plant without agitation will work with ammonium nitrate-kieselguhr in the ratio 3:2 by weight, provided the cooling arrangements in the generator for the absorption period are adequate. There is no difficulty during generation with the absorbent present.

Absorption of NH_3.

This portion of the cycle requires chief consideration. The efficiency of the plant as a whole will depend almost entirely on the proper utilisation of the information contained on Graph II. The rate of absorption will depend primarily on the efficiency of the mechanical arrangements for removing the heat of reaction between the ammonium nitrate and the ammonia. As the pressure rises in the absorber the efficiency of the plant as a whole falls off, for the absorption of heat in the expansion coils depends on the difference of pressure between the liquid ammonia and the absorber pressure. On the other hand an increase of 10 lb. per square inch in pressure of NH_3 in the generator is equal to lowering the cooling water 27° F. (15° C.), and the added rate of absorption and the increased amount of ammonia absorbed may more than compensate for the lowering of thermo-dynamic efficiency.

That the output of the plant may be doubled, due to increase in the ammonia absorbed by using ice water as the final cooling medium, is obvious from the curves, but it is quite possible that the added rate of absorption would add to this increase in efficiency. The gain and loss due to the use of ice water as a cooling agent can be calculated except in so far as relates to the rate of absorption, which can only be experimentally determined on every type of plant.

The density of the kieselguhr-ammonium nitrate mixture gently tapped down was found to be 0·49—a cubic foot of the mixture weighed 494 ozs., and this quantity contained 300 oz. of ammonium nitrate.

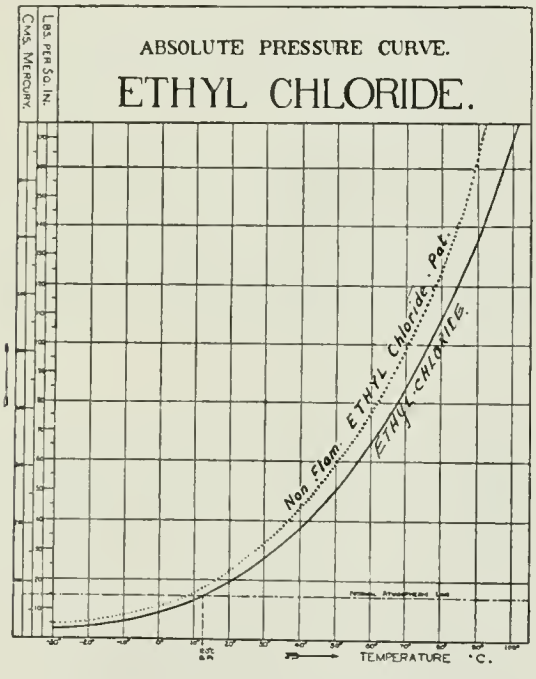
The specific heat of the mixture was found to be 0·24 and the heat conductivity of mixture and components

	K
Kieselguhr	0·00031
Am. nitrate	0·00043
The mixture	0·00036

The ammonium nitrate used passed 1/20 sieve and stopped on 1/50 sieve. Comparison figures are given

K = 0·0004 wood
0·0005 glass
0·0001 wool
0·0001 paper

In order to liquefy 1 lb. of NH_3 gas at 0° C. under pressure about 520 B.Th.U. have to be removed. In the proposed process about 3 lb. of ammonium nitrate are used to absorb 1 lb. of ammonia, and during this absorption the salt is changed from the solid crystalline state to the liquid state. This alteration of state is accompanied by an absorption of about 110 B.Th.U. per pound, so that of the 520 heat units required to condense the 1 lb. of NH_3, 330 are found by the physical change of state of the absorbing body. Similarly in the evolution cycle the separation of the salt in the solid form provides 330 heat units out of the 500 required to gasefy the liquid ammonia. Against this is to be placed the heat of combination of the NH_3 with the NH_4NO_3, but in such unstable bodies this heat of combination must be very small. The figures attained in the Seay trial plant were the best evidence of the theoretical possibilities of the process. The discovery that aluminium sheeting is in no wise affected by either ammonium nitrate or its compounds with ammonia was of vital importance, for steel is rapidly corroded.



Dotted Line = Ethyl Chloride. Non. flam. pat.
For gauge pressure deduct 14.7 lbs. or 76 cm. mercury from above readings.

Graph VIII.

The foregoing research gives information which points to the possibility of constructing a plant with no moving parts whatever. So long as attempts were made to absorb and generate ammonia gas in trays containing ammonium nitrate, so long was success unlikely owing to the formation of solid crusts of porous ammonium nitrate and the difficulty of attaining equilibrium conditions. In a trial plant run on these lines the works experience was in line with the experimental results shown in Graph I. The use of an

absorbing material opens up prospects of overcoming these difficulties.

In view of the growing importance of the production of synthetic ammonia by the Haber process or some modification thereof, and the difficulty of removing the ammonia when formed from the nitrogen hydrogen mixture, attention is drawn to Graph VI., whereon are given the pressure temperature relations of liquid NH_3, ammonium nitrate containing 32% NH_3 and ammonium nitrate with 3·5% NH_3. It will be seen that the vapour pressure of NH_3 in the solid solution is much lower—about ⅛ that of liquid ammonia. As the NH_3 can be driven out of the ammonium nitrate by low-temperature heat, it would appear that use might be made of the reactions in the Haber process.

In the introductory remarks reference was made to the use of ethyl chloride in refrigeration plants for the Navy. The outstanding danger from these plants has been that of fire. It is interesting to note that Mr. Henning has proposed the use of admixtures of methyl bromide with ethyl chloride in such proportions that not only is the vapour non-inflammable, but it is an excellent fire extinguisher.

The mixture of these two bodies, which have boiling points of 4·5° C. and 12° C., are so close together that in practice no fractionation takes place as might have been feared, and the Graph No. VIII. shows the relative pressure temperature curves of ethyl chloride and the methyl bromide-ethyl chloride mixture.

The ability to use the stored cylinders of liquid for the supply of the refrigeration machine as potential fire extinguishers wherewith to flood ammunition chambers is an appreciable advantage.

Our thanks are due to the Seay Syndicate for the generous way in which they have allowed these results to be made public, and also to Mr. Henning for providing the data with reference to the pressure temperature relations of ethyl chloride and its admixture with methyl bromide.

Discussion.

The Chairman asked if Dr. Ormandy could give them more particulars of the freedom from attack of aluminium in ammonium nitrate solutions, as during the war in a lot of work he (the Chairman) carried out he found that aluminium was, under certain conditions, readily attacked by ammonium nitrate.

Dr. Caven said the points he would like to raise were rather academic, and perhaps if curves had been available the matter would have been quite clear. He wished to ask what was the actual nature of the liquid produced when ammonia was absorbed by ammonium nitrate. Did it consist simply of one or more of the compounds Dr. Ormandy mentioned, or was it a solution of ammonium nitrate in liquid ammonia. The reason he asked the question was this. If there was a single curve of dissociation pressure the gaseous phase must be in contact with something quite definite in the state of liquid, and presumably if there were different curves or curves with kinks in them there would be successive stages of definiteness, so to speak, in the liquid phase. He (Dr. Caven) would like to know whether they were really dealing simply with ammonia gas in equilibrium with two compounds, one of which was dissociating producing the other. That would necessarily produce a definite curve with a definite equation.

The other matter had to do with what the Chairman had just mentioned, the question of the action of ammonium nitrate upon aluminium. He (Dr. Caven) was interested a few years ago in some experiments on ammonium nitrate solution. He supposed it was fairly well known that ammonium nitrate solution did not attack aluminium. Ammonium nitrate solution could be evaporated in an aluminium container, whilst it could not be evaporated in an iron container without very great corrosion. He carried out experiments which made it perfectly clear that corrosion was due to hydrolysis of the ammonium nitrate, and the action of the resulting hydrolytic nitric acid on the iron with accompanying loss of ammonia. Much ammonia was lost from ammonium nitrate solution when it was evaporated from iron, and nothing lost when evaporated from aluminium.

There was one other point, viz., that with regard to the mixture of ethyl-chloride and methyl-bromide. It was very remarkable that this mixture was a fire-extinguisher, while ethyl-chloride itself gave rise to the danger of fire. Was there any explanation of that extraordinary fact?

Mr. Stewart said he had been very interested in the paper. It was an example of the application of theory pure and simple before large scale work, and showed that justice should be done to the theoretical side before embarking on large-scale practice.

Mr. Hill said there was one question he would like to ask regarding nitrate of ammonia. Was there any possibility of the mixture gradually separating as the process went on? It occurred to him that this might probably happen, and if it did then the stationary plant would be gradually put out of action, and it would be a very difficult thing to keep it in order.

Dr. Ormandy, replying to the discussion, said it was not necessary for him to answer Mr. Reavell, because the next speaker dealt with the subject. Experience has shown that rolled aluminium which had been turned in the lathe, after two years' use in a plant which was continuously in contact with ammonium nitrate and ammonia, at times free and at times with the ammonia absorbed, did not show the slightest signs of even surface corrosion at any point.

Dr. Caven referred to the nature of the liquid. If one took 50% ammonium nitrate-ammonia compound and heated it gradually the pressure curve was perfectly smooth and could be expressed by a simple equation of the nature of the one given. Investigation showed, however, that the rate of absorption of ammonia varied in a peculiar way with the temperature, and there would appear to be some reasonable evidence for the assumption that the ammonia and ammonium nitrate really did tend to form a series of compounds as the curves illustrating the rate of absorption made pronounced changes in curvature at or about the points corresponding to comparatively simple molecular ratios.

The next point raised was regarding the price of methyl-bromide. He (Dr. Ormandy) thought experiment had shown that methyl-bromide was four times as efficient as carbon-tetrachloride for extinguishing a type of fire which was exceedingly difficult to put out by any other means, and that was the inflammation of a highly volatile liquid such as petrol, benzol, or the like, and he did not think that one-fourth of the bulk of methyl-bromide to-day, assuming that it was four times as efficient, would cost necessarily more than the larger bulk of carbon-tetrachloride. One of the advantages that arose from the use of methyl-bromide was the fact that it boiled at such a low temperature, and if it were in a vessel at ordinary temperature it would be present in sufficient pressure to expel itself at considerable velocity and thus enable a very simple type of fire-extinguisher to be made. It needs no pressure generating apparatus with it the whole time, and, moreover, whether the vessel is half

full or one-third the pressure remains constant. It would be foolish to suggest methyl-bromide for use on a large scale, but only in dealing with incipient fires.

Regarding the effect of constant absorption and emission from the ammonium nitrate-ammonia mixture, many experiments were made to find out what effect the constant absorption and expulsion of ammonia would have on the physical nature of the ammonium nitrate kieselguhr absorbent. After many hundreds of alternations not the slightest difference could be found in the ordinary or microscopic appearance of the absorbing body, and there was every reason to believe that the process could be continued indefinitely.

In view of the growing use of the Haber process for producing synthetic ammonia or the modifications of this process, it was interesting to consider the possible application of ammonium nitrate to recover the ammonia out of the mixture of hydrogen and nitrogen. The ordinary Haber process gave a mixture containing only 7 per cent. or 8 per cent. of ammonia gas in the hydrogen nitrogen mixture. In the early stages efforts were made to remove this ammonia by a cooling process with liquefaction of the gas, but great trouble was experienced, and later on water absorption methods were employed. In view of the fact that ammonium nitrate will absorb ammonia with great rapidity, and will reduce the vapour tension at any temperature to about one-eighth that of ammonia gas in relation to liquid ammonia at that temperature, it would appear a practicable scheme to make use of ammonium nitrate as a means for removing the ammonia from the gas whilst under pressure.

Dr. Ormandy said he would like to thank the Seay Syndicate for kindly allowing him to make the experiments public, because the work was carried out for them; and on behalf of Mr. Caven and himself he (Dr. Ormandy) would also express thanks to the Glasgow Section, and to Mr. MacGregor in particular, for the zeal he had shown in getting the meeting together, and to Mr. Reavell for so kindly coming up to take the chair.

The meeting closed with a hearty vote of thanks to Dr. Ormandy for his paper.

FIFTH CONFERENCE.

Subject :

"EVAPORATION AND DISTILLATION."

The meetings of the Fifth Conference of the Group were held in the Institution of Shipbuilders and Engineers, 39, Elmbank Crescent, Glasgow, on the occasion of the Annual General Meeting of the Society of Chemical Industry.

Afternoon Session: WEDNESDAY, JULY 6, 1922.

Chairman: W. H. COLEMAN, Esq.

The first paper read was

OBSERVATIONS ON THE DESIGN OF AMMONIACAL LIQUOR STILLS.

By P. PARRISH, A.I.C.

It is not proposed in this paper to enter into a consideration of the relative merits and demerits of direct-fired intermittent stills for the distillation of ammoniacal liquor. It will be assumed that sound scientific and technical reasons justified the abandonment of such plant.

The origin of the present type of continuous columnar still for the distillation of gas liquor can be traced to a Frenchman of the name of Savalle, who was responsible in 1875 for many patents which aimed at improving the apparatus and processes for the distillation, purification, and rectification of alcohol.

Apparently Savalle was intimately conversant with the design, and had had practical experience of the apparatus invented by Aeneas Coffey in 1831 for producing, in one operation, a more concentrated spirit from alcoholic mashes and at the same time removing, to a large extent, the objectional by-products.

As the Coffey still, a sketch of which is shown below, is typical of many stills commonly in use in this country, it is proposed to describe it in some detail to afford an insight into the principles of continuous working. The still is constituted of two columns, an analyser, and rectifier. The analyser comprises a series of chambers which are separated by perforated copper plates, each having a safety valve and an overflow pipe, one end of which is arranged to give a liquor level of about 1½in. on the plate, the other side dipping into a sealed cup. The overflows are alternately placed, as shown.

In starting the apparatus, steam, which is introduced to the analyser, passes through the perforations of the several plates, and is led to the foot of the rectifier. When the two latter pieces of apparatus have reached a suitable temperature the wash is pumped from the wash tank and enters a pipe at the top of the rectifier. This pipe is a continuous one, and passes through the latter column and is carried to the analyser, where the wash is discharged. The wash, whilst cooling the steam and alcoholic vapour stream, is simultaneously preheated in its passage through the rectifier. The perforated plates presumably are designed on the basis of a definite vapour velocity which admits of the ascent of the steam, but ob-

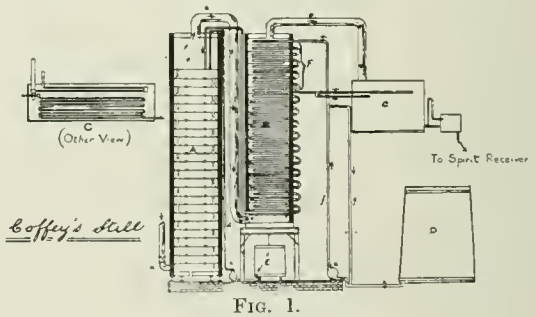


FIG. 1.

Coffey Still.

KEY.

A. Analyser.
B. Rectifier.
C. Condenser.
D. Wash Stock Tank.
E. Hot Feints Vessel.
F. Finished Spirit Condenser.

a. Steam inlet.
b. Alcohol vapour: steam outlet.
c. ,, ,, ,, inlet.
d. Weak spirit: return pipe.
e. Alcohol vapour: steam pipe.
f. Wash pipe.
g. Wash return pipe.
h. Feints centrifugal pump.
h. Wash ,, ,,

viates the passage of the wash. The liquor must travel across the whole area of the plates and descend through the overflows provided. Thus the descending wash meets the ascending current of steam, the bubbling action so produced promoting the release of the alcohol. The alcoholic vapours and steam pass away from the top of the analyser to the foot of the rectifier, bubbling through the reflux liquid formed by the partial condensation of the vapours owing to the cooling influence of the wash. With the passage of the vapours upwards through the rectifier, they become richer in alcohol, the descending reflux liquid becoming weaker in spirit, but retaining the less volatile fusel oils.

At a certain part of the rectifier the temperature obtaining in which coincides with the temperature of the strong alcohol condensate, an unperforated copper plate is fitted having a wide pipe which

be said that these two liquors are identical, and it is not inconceivable that the absence of any marked advance in the design and arrangement of plant for the distillation of ammoniacal liquor in the production of ammonium sulphate is, in a degree, attributable to the assumption, which appears to have been perpetuated through several decades, that the physico-chemical principles underlying the distillation of the two liquors were the same.

The quantity of gas liquor produced and distilled in the United Kingdom must be of the order of 1250 million gallons of 8 oz. (1·73 per cent.) NH_3 per annum. This quantity chiefly arises in the purification of crude coal gas derived from the carbonisation of coal at gas works and coke ovens, from blast-furnace gases, and the carbonisation of shale as conducted largely at the shale oil works in Scotland.

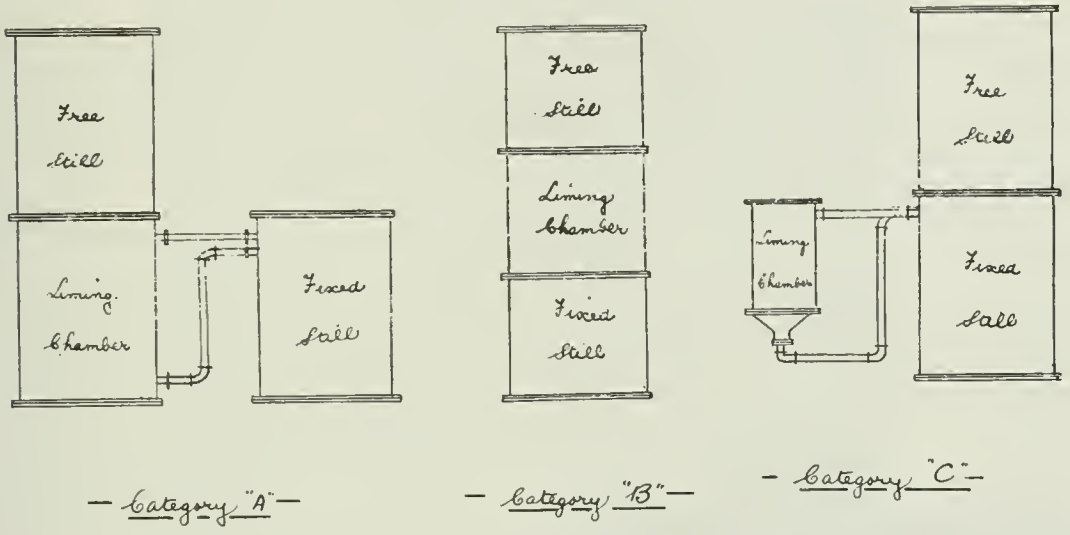


FIG. 2.

Showing arrangement of distillation plant in three categories.

projects above the plate to the extent of about 2in. This "spirit plate," as it is called, is drained by a two-branched pipe. Under normal working conditions the alcohol condenses above the spirit plate and runs through one branch to the condenser and away to the spirit receiver.

Distillation proceeds uninterruptedly for two or three days, at the beginning and end of which weak spirit condenses and is run to the condenser and thence to the "hot feints" vessel below. The reflux liquid, containing the alcohol and fusel oils, also passes to the "hot feints" vessel and is pumped continuously to the analyser and is redistilled. The weak spirit vapours escaping at the top of the rectifier are condensed and also led to the "hot feints" vessel, from which the fusel oils are removed at the end of each distillation period.

Savalle's inventive genius was directed to the improvement of the Coffey still and accessory plant, and presumably, by reason of his achievements, his attention was turned to the necessity for an improvement of the continuous columnar still for the distillation of ammoniacal liquor. Here is an instance, not uncommon in the chemical industry, where the experience and practice of one industry, due to the seeming analogous conditions, is applied to the service of another.

Whilst there are points of similarity in the distillation of wash and ammoniacal liquor, it cannot

The following analyses give an indication of the average composition of ammoniacal liquors arising at various works:—

	Gas works. % w/v.	Coke oven. % w/v.	Blast furnace. % w/v.	Shale works. % w/v.
Volatile ammonia..	1·4 to 2·5	0·68 to 0·85	0·4	0·9
Fixed ammonia ..	0·1 to 0·4	0·3 to 0·10	0·01	0·3
Total ammonia ..	1·5 to 2·9	0·98 to 0·95	0·41	0·93
$(NH_4)_2S$	0·8 to 0·9	0·40	—	0·10
$(NH_4)_2CO_3$ ..	5·0 to 8·5	1·90	1·1	2·90
NH_4Cl.	0·5 to 1·0	0·20	0·006	0·015
NH_4CN	0·07 to 0·3	0·04	—	—

The gas liquor as produced in Scotland, generally speaking, contains about 10 per cent. of the ammonia as fixed ammonia, as compared with about double the quantity which gas liquors as produced in England contain.

The gas liquor with which the author has had to deal during the last few years has exhibited a remarkably constant CO_2: H_2S ratio, viz. 7: 1, whilst the NH_3: H_2S ratio has averaged 1.73 per cent.: 0.28 — 0.35 per cent.

The ammonium compounds constituting gas liquor are divided under two heads:—(1) *Those volatile at ordinary temperatures*, viz., ammonium monocarbonate, bicarbonate, sesquicarbonate, hydrosulphide, sulphide, and cyanide. (2) *Those*

fixed at ordinary temperatures, but which can be decomposed by an exchange of alkali base: Ammonium sulphate, sulphite, thiosulphate, thiocarbonate, chloride, thiocyanate, ferrocyanate. Gas liquor also contains pyridine bases, phenols, naphthalene, and tarry matter, which vary appreciably according to the extent to which the liquor is separated from the tar in the "wells" at the

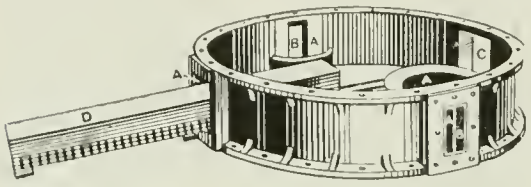


FIG. 3.
Cleaning arrangement.

gas and coke oven works, and whether or not it is filtered to eliminate the floating tar or hydrocarbons.

Gas liquor containing tarry matter is disadvantageous in many ways, and too much stress cannot be laid on the necessity for efficient separation of the tar from the liquor and for the adoption of similar methods for the removal of the hydrocarbons specifically lighter than the liquor itself.

It is not proposed to enter into a lengthy discussion of the methods for the analysis, or evaluation, of gas liquor. This subject has been admirably dealt with in the last few years in the annual reports of the Chief Alkali Inspectors, and the work of Mr. Linder in this connection is well known.

Many gas works in England still state the content of ammonia in terms of oz. strength, which originally implied the numbers of ozs. of real sulphuric acid required to saturate 1 gall. of the gas liquor. This was the basis of the Wills

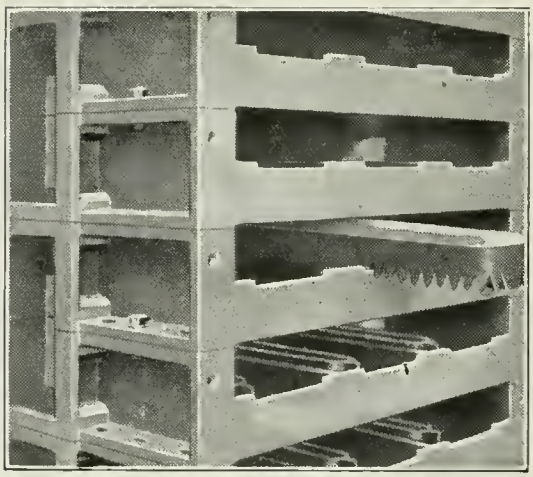


FIG. 4.
Cleaning arrangement.

saturation test, but as it did not contemplate the fixed ammonia, it is now usual to pursue one or other of two methods to arrive at the total content of ammonia.

At some works it is usual to distill off the free and fixed ammonia of the gas liquor into acid which, at 15° C. contains 10 parts by weight of real sulphuric acid in 100 parts by volume, titrating from a burette of 1 oz. capacity graduated in 1/16ths of an oz., and subdivided into tenths (*i.e.*, into 1/160ths in all), and to read off the oz. strength direct. In other cases, the free and fixed ammonia content of the gas liquor is distilled into N/10 sulphuric acid, and the ammonia content expressed in terms of percentage of ammonia w/v and a factor used for conversion to oz. strength.

The following data will doubtless be useful as bearing on the oz. strength in its relationship to percentage of ammonia:—

(1) $2NH_3 + H_2SO_4 = (NH_4)_2SO_4$
34 98 132
0·3469 oz. 1 oz. 1·3469 oz.

(2) 1 oz. strength = 0.3469 oz. of ammonia per gallon.

(3) As 1 gall. contains 160 oz. therefore the parts of ammonia by weight per 100 parts by volume will be:
160 : 100 : : 0.3469 : x
$x = 0.217$.

(4) If 0.217 part of ammonia by weight per 100 parts by volume represents 1 oz., then the factor for converting per cent. ammonia w/v to oz. strength is: $1.0 \div 0.217 = 4.61$.

(5) In London and the South of England liquor is still sold by, or expressed in terms of, butts of 108 gallons.

(6) One degree Twaddel is usually regarded as equal to 2 oz. strength.

(7) To arrive at the number of butts of liquor (say) of 8 oz. strength required to produce 1 ton of sulphate of ammonia of 25.25 per cent. ammonia content.
(a) One ton of sulphate of ammonia of the above content will contain 0.2525 ton of ammonia, or 9049.6 oz. of ammonia.
(b) One gallon of 8 oz. liquor contains 2.7752 oz. of ammonia.
(c) $9049.6 \div 2.7752 = 3260.8$ galls. per ton.
(d) $3260.8 \div 108 = 30.192$ butts per ton assuming 100 per cent. yield.

(8) To arrive at the number of tons of gas liquor required to produce one ton of ammonium sulphate of given content, divide the percentage of ammonia in the sulphate by the percentage of ammonia in the liquor. Assume 8 oz. liquor (*i.e.*, $8 \times 0.2168 = 1.734$ NH_3) and 25.25 per cent. NH_3 as the content of the sulphate of ammonia.
$25.25 \div 1.734 = 14.562$ tons,
or $14.562 \times 224 = 3261.8$ galls.,
or $3261.8 \div 108 = 30.192$ butts.

Table showing tons of gas liquor of varying strengths required to produce one ton of Ammonium Sulphate, having varying contents of ammonia.

Strength of gas liquor.		Tons of gas liquor per ton of ammonium sulphate based on 100% conversion efficiency.							
		Ammonia content of sulphate of ammonia.							
% ammonia.	Oz. strength.	25 %	25·1 %	25·2 %	25·3 %	25·4 %	25·5 %	25·6 %	25·7 %
0·868	4	28·90	29·02	29·14	29·25	29·36	29·47	29·59	29·71
1·085	5	23·03	23·13	23·23	23·31	23·41	23·50	23·59	23·68
1·302	6	19·21	19·29	19·36	19·44	19·51	19·59	19·66	19·74
1·519	7	16·46	16·53	16·59	16·65	16·73	16·79	16·86	16·92
1·730	8	14·45	14·51	14·56	14·62	14·69	14·74	14·80	14·86
1·953	9	12·80	12·85	12·90	12·95	13·00	13·05	13·11	13·16
2·170	10	11·52	11·57	11·61	11·66	11·70	11·75	11·80	11·84
2·389	11	10·46	10·51	10·54	10·59	10·63	10·67	10·71	10·75
2·604	12	9·60	9·64	9·68	9·71	9·75	9·79	9·83	9·87

Broadly speaking, the combined arrangement of free and fixed stills and liming chamber can be divided into three categories, as illustrated in the

diagrams subjoined, which, it is hoped, are self-explanatory. (See Fig. 2.)

As to the arrangement which the plant should take, much depends on the provision made for cleaning the stills, on the fixed ammonia content of the gas liquor to be distilled, and on its freedom from tarry matter. By far the greater number of stills operating in the United Kingdom are stills built up of double-flanged sections, each of which, generally speaking, constitutes a chamber. The joints between the corresponding flanges of the chambers are made with boiled linseed oil and Vulcan cement, or with red and white lead and lead wire. Obviously these sections have to be provided with such a number of manholes, and of such a size, as will afford facilities for the cleansing of the overflow pipe or pipes and the removal and/or cleaning of the hoods, frogs, crocodiles, or whatever steam-distributing device is provided. Figs. 3 and 4 show typical arrangements such as are usually made in this connexion.

Two disadvantages accompany the use of this form of still, irrespective of any disability which may arise by reason of the category, as above defined, under which it comes. The joints between the several sections are potential sources for the leakage of ammonia, and as it is unusual to insulate the covers of the manholes with non-conducting material, the loss of heat by radiation is not inconsiderable. It has to be remembered, with the type of still under review, that complete dismantling may become necessary owing to accumulations of lime sludge, either causing blockages which are difficult, if not impossible, to remove *in situ*, or the lime may form laminations on the bottom of the chambers, to such an extent as to seriously impair the thermal efficiency and capacity of the plant.

Another arrangement of still is possible, which largely overcomes the disabilities to which reference has been made.

Figure 5 represents the alternative form, which comprises an outside shell free from a multiplicity of flanges, and without cleaning manholes. This

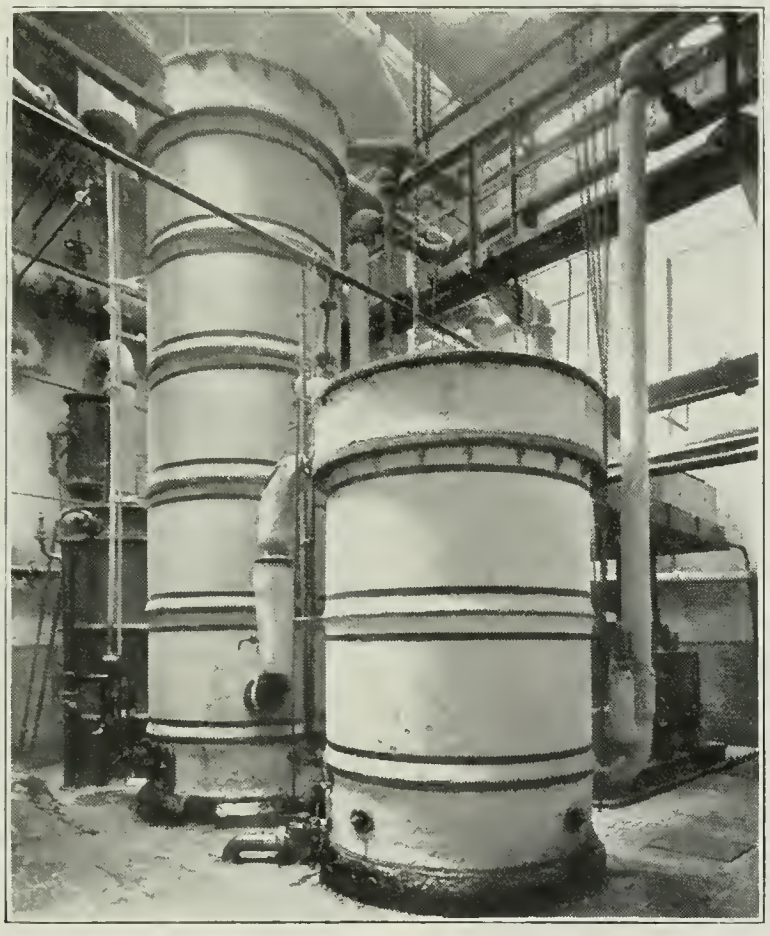


Fig. 5.

Ammoniacal liquor still (Free and Fixed).

design of still, for which Dr. Carpenter was responsible, contemplates the withdrawal of the internal elements, which, as shown in Figs. 6 and 7, comprise a hood, tray, and bubbler, as constituting a complete chamber for cleaning purposes.

These hoods, trays, and bubblers are provided with machined faces so that there is no possibility of the by-passing of steam on the outer part of the trays. Suitable lifting tackle, as seen in Fig. 8, is provided for facilitating the removal of the internal elements, which, in turn, are all provided with suitable lifting lugs.

It has been found in practice that, provided the accumulations of lime sludge at the foot of the

liming chamber are removed every fortnight, and the top cover of the fixed still is lifted monthly for the cleaning of the perforations of the anti-priming device, the still will operate continuously for six months before cleaning is necessary. During this time approximately 5,000,000 galls. of

Fig. 6.
Hood and Bubbler.

gas liquor will have been distilled, still cleaning representing a charge of about 2d. per ton of sulphate made. With the shell type of still, involving as it does the withdrawal of the internal elements for cleaning purposes, it would be obviously unwise to arrange the plant according to category B. (*cf. supra*), as this would necessitate

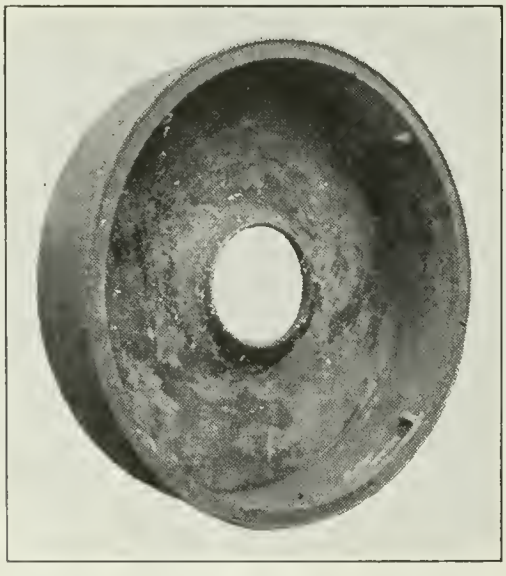


Fig. 7.
Tray.

the removal of the liming chamber in order to afford access to the anti-priming arrangement, or first tray, of the fixed still.

Figure 9 represents a gas liquor still as installed at the Provan Chemical Works of the Glasgow Corporation. The fixed ammonia content of the gas liquor in this case only represents about 10 per cent. of the total ammonia.

Manifestly with gas liquor having a high content of fixed ammonia, particularly if such fixed ammonia is due to ammonium sulphate, the difficulty of keeping the distilling sections of the fixed still free from sludge, particularly the first and second fixed sections, is great. Special consideration should be given to the design of the liming chambers or the elements of large liming chambers, which admit of considerable time contact, in order to ensure active agitation. Easy means of a periodic discharge of the sludge from the foot of the liming chambers should also be provided. Failing this, an arrangement of plant with outside liming section specially designed as represented in category C. is necessary.

It is the author's view that mechanical considerations, such as facilities for cleaning, provision for the removal of bubbling hoods and initial capital cost, have been the factors largely governing the design and arrangement of gas liquor stills in the past. Had designers and makers of gas liquor stills been in a position to visualise the items which contribute to the cost of ammonium sulphate in so far as the still itself is concerned, it is not improbable that less attention would have been

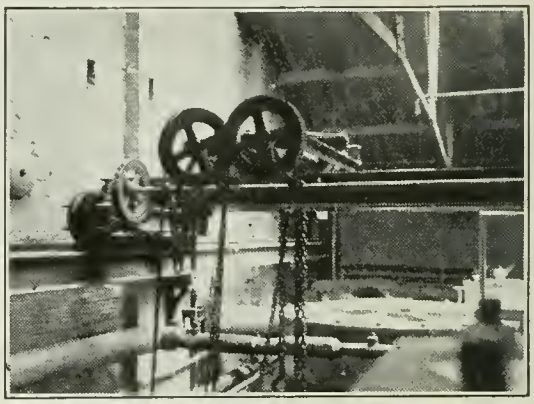


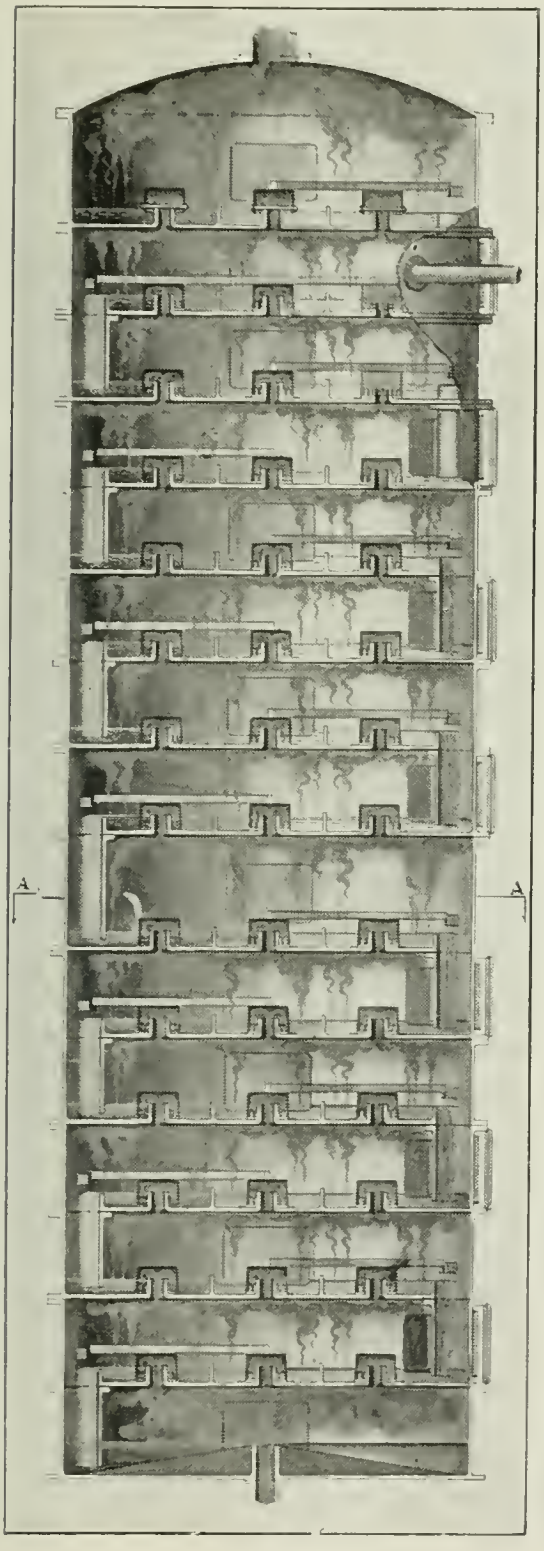
Fig. 8.
Lifting tackle for shell type of still.

directed to this somewhat circumscribed aspect of design, and more attention given to the possibilities of designing a still which would be economic as regards steam consumption. In saying this, it is not overlooked that the ideals of the chemist and physicist have not infrequently to be compromised in order to ensure mechanical strength and practicability in the matter of production.

The data given below relative to the characteristics of several types of present-day stills are impressive in many ways, and will repay careful study. Whilst recognising the various local conditions under which stills have to operate, and the variation of the quality of liquor with which they will be called upon to deal, one is led almost irresistibly to the conclusion that standardisation based on experimental data under varying conditions, and with due regard to the fundamental laws governing the distillation of gas liquor, would be fully warranted.

Solutions of ammonia of such strengths as are typical of normal gas liquor behave generally according to Henry's law. This law provides that the vapour pressure (P) of a volatile solvent and solute is expressed by $P = ax$, in which a is a constant and x the molecular fraction of the solute in relation to the solvent. In the case of ammonia

solutions a for accurate values has to be experimentally determined by ascertaining P either by chemical analysis of the vapour over a solution, or by the static method of determining vapour pressure.



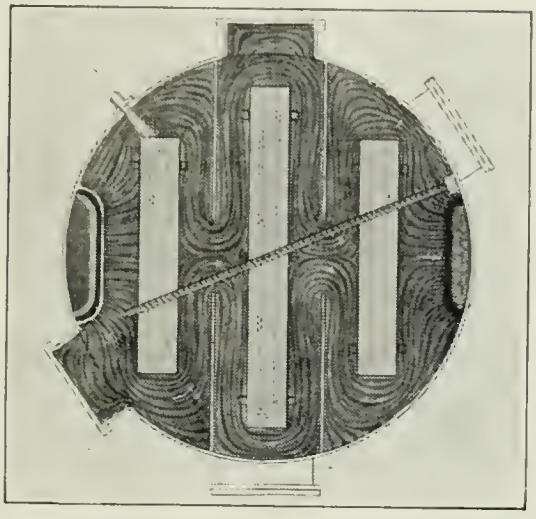


FIG. 9.

Ammoniacal liquor still, as installed at the Provan Chemical Works of the Glasgow Corporation.

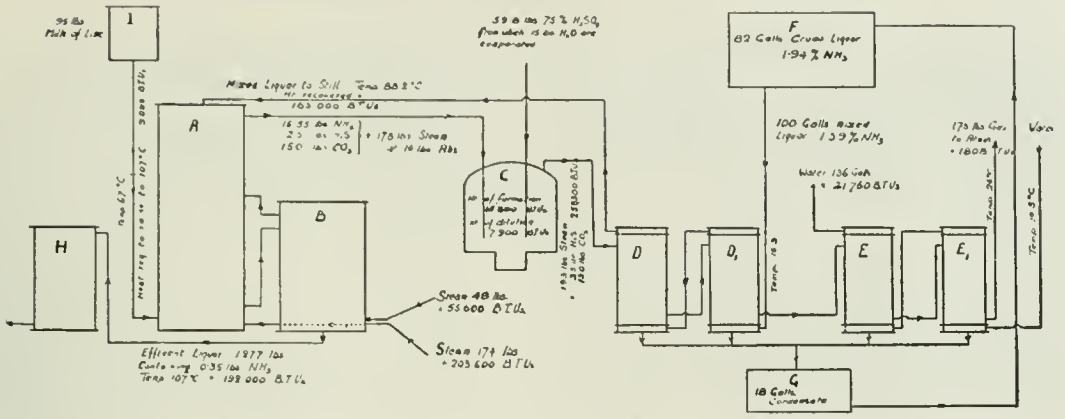
For ammonia and water the values of a are well-known ammonia being of immense industrial importance in the manufacture of soda ash by the ammonia-soda process. The values show, however, a slight deviation from those as calculated by Henry's law, owing to the ammonia chemically combining with the solvent to form an easily dissociated ammonium hydrate. In addition, the deviation of experimental and calculated values increases with decreases of concentration. Such deductions as may be made from the above law apply of course only to conditions in a certain section of the gas liquor still, probably the lower portion of the free still, where, owing to the previous decarbonisation and desulphurisation, the liquor assumes that of an ordinary ammonium hydrate solution. Although the gas laws allow of a ready interpretation of conditions obtaining in the still, and indeed assist in affording certain fundamentals of design, a point is reached at which further progress in this direction can only result from patient observation of details such as can only be obtained from practical working conditions. A consideration of the costings of ammonium sulphate manufacture will reveal that the labour involved in the control and operation of the still does not represent more than 4 per cent. of the total cost of manufacture, that the cost of cleaning represents 0·2 per cent. of the cost of manufacture, whilst a figure approaching 10 per cent. more nearly constitutes the cost of the steam used in the distillation of a normal gas liquor, quite apart from the steam used for motive purposes. These figures have reference to plant operating on a comparatively large scale. Little wonder, therefore, that one's attention is directed to the thermal aspect of this manufacture, concerning which singularly little information, or data, appears to have been published. With a view to constructing a thermal balance sheet, a sulphate of ammonia still operating under ordinary working conditions was selected as the source from which the following measurements were made:—

(1) Quantity of liquor distilled.
(2) Quantity of steam supplied.
(3) Temperature of steam supplied.
(4) Pressure of steam supplied.
(5) Quantity of devil liquor distilled.

Characteristics of ammoniacal liquor stills as supplied by English makers.

Letter index.	Diameter of free still.	Height of free still.	Diameter of fixed still.	Height of fixed still.	No. of trays in free still.	No. of trays in fixed still.	Depth of liquor in trays.	Liquor capacity of trays (galls.).	Depth of liquor seal.	Heating surface per tray or element.	Capacity per hour (galls.).	lb. of NH_3 per hour.	Surface area occupied by hoods, crocodiles or other such devices.	Depth of chamber.	Arrangement of overflow device.	Arrangement for cleaning.	Arrangement at top of still to avoid entrainment.	Surface area for ascent of gases.	Surface area for descent of liquor.	Weight of fully equipped still.	Letter index.
	ft. in.	ft. in.	ft. in.	ft. in.			in.		in.	sq. ft.			sq. ft.	ft. in.				sq. in.	sq. in.	tons.	
A.	5 10	21 10	5 10	10 2	6	5	5	57·25	1	21·59	1250	250 (2% NH_3 liquor)	12·48	1 4½	Annular space round hood.	Still to be dismembled.	Anti-priming device.	71·6	278	41	A.
C.	4 0	16 2½	4 0	9 11	10	8	3½	17·62	1 5/16	9·68	1050	182	4·9	11	Two semi-elliptical slots.	Large manholes.	Void.	183	224	22	C.
G.	7 6	18 8	7 6	11 5	9	8	2	43·44	1	41·7	1360	235 (1·73%)	11·6	1 0½	Eight 4″ pipes.	Manholes	Void.	248	59·5	—	G.
I.	5 6 square.	17 7	5 6 square.	7 0½	12	8	2½	41·2	1½	29·15	—	—	3·9	9	One 8″×3″ opening.	Hand-holes.	Large void.	110	48	—	I.
J.	5 0	19 8	5 0	8 11	9	8	2⅝	21·2	1	17·04	—	—	5·37	10	Two semi-elliptical slots.	Hand-holes.	Void.	174·7	180	—	J.
L.	4 6	7 6½	4 6	7 6½	13	8	1 1/16	12·5	1½	12·54	406	70·2 (1·73%)	6·09	6	Six 4″ diameter pipes.	Six handholes 6″ — 3″ for each chamber.	Void.	217·5	73·75	15	L.
Category "B."																					
B.	5 0	12 6	Comprising a conical base with circular steps increasing in dia.	6 8	14	9 steps.	1½	12·75	1½	19	650	141 (2·17%)	11	7½	Two 4″ pipes.	Manholes.	Anti-priming hood.	78·5	14	25	B.
F.	7 0	12 0	7 0	12 0 inclusive of liming chamber.	8	6	3½	58·1	1½	32	2916	437·4 (1½%)	11·53	1 6	Two semi-elliptical slots.	Large manholes.	Void.	373	522	19	F.
H.	6 0	4 0	6 0	6 6	6	8	2	28·0	1	26·9	840	182·2 (2·17%)	12·2	7½	One 8″×4″ semi-elliptical pipe.	Hand-holes.	Void.	113	57	17½	H.
Category "C."																					
D.	5 4	9 0	5 4	12 8	5	7	5	49·5	2	18·96	1250	195 (1·56%)	5	1 3	Two semi-circular pipes.	Manholes.	Void.	213	204	—	D.
E.	5 6 square.	4 0	5 6	8 6	4	8	2½	39·3	1⅝	26·25	560	56 (1%)	11·6	9	Two 12″×4″ openings.	Manholes.	Void.	528	54	—	E.
K.	5 6 square.	8 0	5 6	13 0	10	15	2½	36·4	1½	28·0	860	126 (1½%)	11·6	9	Four 7″×3″ openings.	Manholes.	Void.	528	84	26	K.

Fig. 10.



A Free ammonia still E and E_1 Coolers B Fixed ammonia still F Crude liquor storage
C Saturator G Devil liquor storage D and D_1 Liquor preheaters H Boiler feed heater I Lime feed

FIG. 11.

Summarised heat balance of sulphate of ammonia manufacture.

Dr.		B.Th.U.'s	*Cr.*	B.Th.U.'s.
(*a*) Heat entering still—			Heat leaving still—	
I. Via steam		259,100	I. Via gas stream	241,000
II. „ liquor		165,000	II. „ effluent	192,000
III. „ lime		12,000	III. Radiation loss (0·7%)	3,100
		436,100		436,100
(*b*) Saturator balance				
I. Heat entering		241,000	Heat entering preheaters	258,300
II. Ht. of dilution		7,900	Loss (radiation) via liquor whilst centrifuging, etc. (17·8%)	56,400
III. „ „ formation		65,800		
		314,700		314,700
(*c*) Interchange balance—				
Entering preheaters		258,300	Heat recovered via liquor	165,000
			„ lost via water (9·0%)	21,760
			„ „ „ gas (0·1%)	180
			Radiation losses (21·0%)	71,360
		258,300		258,300

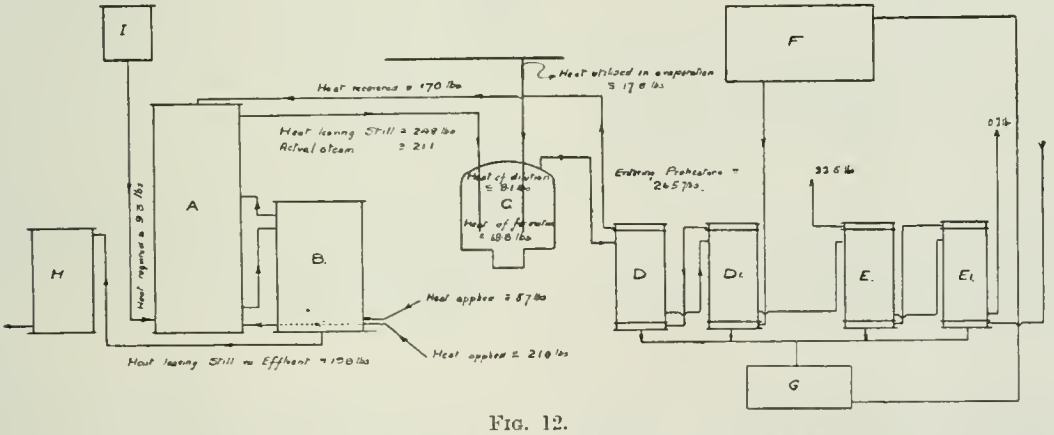


FIG. 12.

Thermal balance of sulphate of ammonia manufacture. No. 2 diagram.
Showing all heat equivalents reduced to pounds steam from and at 212° F.

SUMMARISED HEAT BALANCE.

Dr.		lb.	*Cr.*	lb.
(*a*) Heat entering still.	I. Via steam	267	Heat leaving still.	I. Via gas stream 248
	II. Via liquor	170		II. Via effluent 198
	III. Via lime	9·3		III. Via radiation loss 0·3
		446·3		446.3
			Heat entering preheaters	265·7
(*b*) Saturator balance.	I. Heat entering	248	Loss " radiation " *via* liquor whilst centrifuging, etc.	59·2
	II. Heat of dilution	8·1		
	III. Heat of formation	68·8		324·9
		324·9	Heat recovered via liquor	170
			Heat lost via water	22·5
			Heat lost via gas	0·2
(*c*) Interchange balance.	Entering preheaters	265·7	Radiation losses	73
		265·7		265·7

(6) Quantity of cooling water used.
(7) Temperature of water at the inlet and outlet of the coolers.
(8) Temperature of gas liquor at the inlet and outlet of the preheaters.
(9) Temperature and quantity of cream of lime used.
(10) Temperature and quantity of effluent liquor discharged from the still.
(11) Temperature of waste gases leaving the coolers.
(12) Ammonia, hydrogen sulphide, and carbonic acid content of crude liquor distilled.
(13) Ammonia, hydrogen sulphide, and carbonic acid content of the gases leaving the still in their passage to the saturator.

These data were collated and reduced to a definite basis of one hundred gallons of combined gas liquor and devil liquor distilled.

Owing to the heat of formation of ammonium sulphate by the combination of the ammonia with the acid of the saturator, the temperature of the bath is at least equal to the temperature of a boiling saturated solution of ammonium sulphate. A further increase of temperature accrues from the free sulphuric acid content of the bath. Indeed the temperature of the bath will be at least 15° C. higher than the temperature of the stream of steam and ammonia leaving the still. Hence there can be little possibility of the condensation of water in the saturator unless the heat of formation of ammonium sulphate is counterbalanced by heat losses *viâ* conduction, radiation, and the circulation of mother liquor outside the saturator.

The factors governing the water balance of a saturator are undoubtedly the strength of acid which is used and the quantity of extraneous water which is introduced thereto.

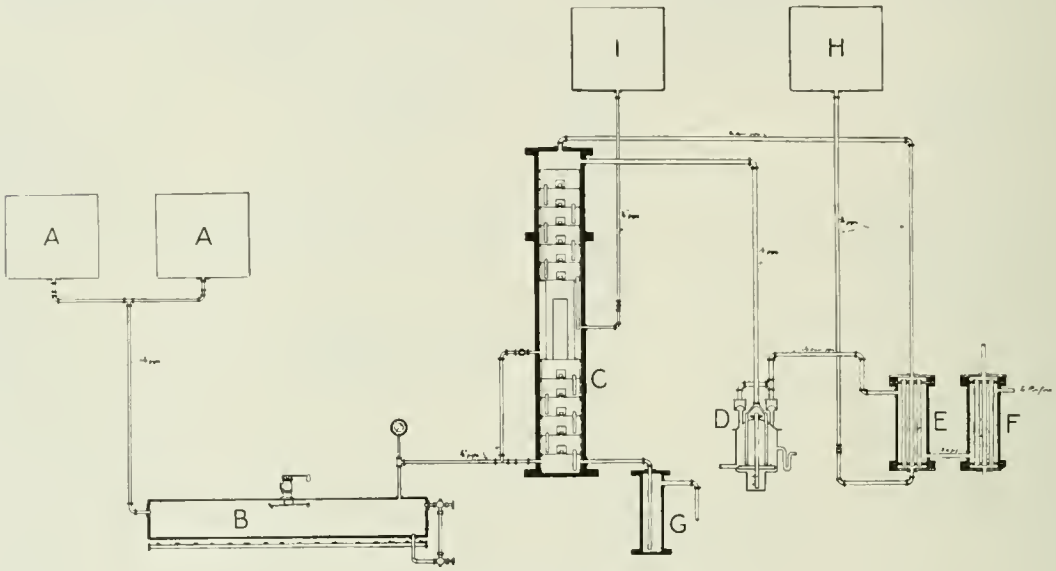


FIG. 13.

Complete experimental sulphate of ammonia plant.

A. *Water feed tanks*, 20 in. dia., 18 in. high.
B. *Boiler.* Constructed of 8 in. pipe, 5 ft. long, with gauge glass, safety valve, and pressure gauge; heated by gas jets.
C. *Combination still*, 5 ft. 8½ in. high, 9 in. dia., with twelve trays 8½ in. dia. by 4 in. high, and liming chamber 1 ft. 4 in. high by 3 in. and 6 in. dia.
D. *Saturator.* 9 in. dia., 9 in. high, 4 in. dia. well with two catch boxes, 3 in. dia. 3 in. high, and ¾ in. pipes.
E. *Heater* of 6 in. pipe, 2 ft. high with seven 1 in. pipes inside with making-up pieces as shown.
F. *Cooler* of 6 in. pipe, 2 ft. high with seven 1 in. pipes inside with making-up pieces as shown.
G. *Seal pot*, 4 in. dia., 2 ft. high.
H. *Gas liquor tank*, 20 in. dia., 18 in. high.
I. *Cream of lime tank*, 20 in. dia. and 18 in. high.

Diagram 1, Fig. 11, shows the heat expressed in terms of B.Th.U. entering and/or leaving the various parts of the plant constituting a complete sulphate of ammonia unit, and diagram 2, Fig. 12, shows for purposes of easy comparison the same determinations expressed in pounds of steam from and at 212° F. In each diagram a summarised heat balance has been drawn up showing the thermal aspect as affecting (*a*) distillation; (*b*) the saturator; and (*c*) the preheaters and coolers.

It is not proposed at this juncture to discuss the summarised heat balance, except to deal with one aspect which has become increasingly prominent with many technicians by reason of the development in the production of neutral sulphate of ammonia by processes which necessitate the use of a washing medium for effecting the neutralisation of the acid-containing sulphate of ammonia.

Not infrequently has the question been asked, What are the conditions governing the water balance of a saturator?

It is known that when using sulphuric acid of 70 per cent. H_2SO_4 content, 50 gallons of extraneous water can be introduced to the bath without creating redundant mother liquor.

From the thermal data afforded by the measurements under review a simple calculation will show that 55 gallons of water were being evaporated per ton of ammonium sulphate (of 25·74 per cent. strength) being made.

So far it will be seen that there is great uncertainty as to the form which the elements of an ammoniacal liquor still should take if the vital factor, namely, steam consumption within the still, is to be economised. Hitherto hypotheses have been deduced and indeed worked upon, but it is doubtful whether any satisfactory data have been arrived at, either experimentally or otherwise, to indicate the precise form which the elements should assume, and the conditions under which the stills should be operated if a minimum steam consumption is to be attained.

Having an experimental sulphate of ammonia plant available of the size and capacity as indicated in Fig. 13, experiments were conducted under varying conditions with a view to arriving at certain conclusions in the above connexion.

Figure 14 is a diagram showing the liquor and gas concentrations at two rates of feed with identical steam consumption. It will be seen from this

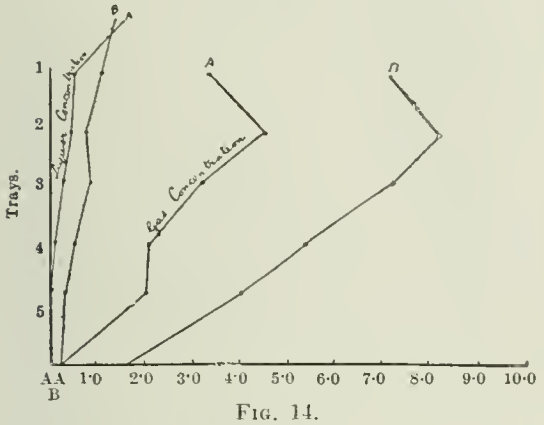


FIG. 14.

Diagram showing liquor and ammonia gas concentrations at two rates of speed.

3·7 *galls. per hour A* } *with identical steam consumption.*
9·3 ,, ,, ,, *B* }

diagram that the rate of flow has been increased by about 250 per cent. at the sacrifice of 0.23 per cent. of free ammonia in the effluent. It is not inconceivable that an identical effluent to that of the lower rate of feed would have been obtained had the time contact been increased (say) to the extent represented by two additional trays. This experiment would appear to indicate that given a definite time contact, the steam consumption can be made to approach the theoretical minimum possible dependent upon the liquor feed concentration. The latter condition embodies the ideal at which designers of gas liquor stills should aim, and incidentally the one with which manufacturers should be conversant when contemplating the purchase of gas liquor stills.

A measure of the efficiency of the steam consumption of a gas liquor still is afforded by the

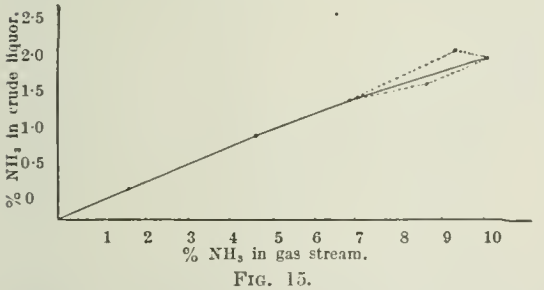


FIG. 15.

Diagram showing ammonia concentration of gas, with varying contents of ammonia.

concentration of the ammonia in the gas stream. A simple method of determining this factor under practical working conditions is to condense the stream of steam and ammonia leading to the saturator, and to make a determination for ammonia, carbonic acid, and hydrogen sulphide contents. From a series of analyses carried out in this manner, the graph shown in Fig. 15 has been prepared. By calculation from the foregoing graph the curve in Fig. 16, which affords an indication of the steam consumption per lb. of ammonia in relation to the ammonia concentration in the crude liquor, has been constructed.

Unfortunately little information appears to be available concerning the extent to which the concentration of ammonia in the gas stream is affected

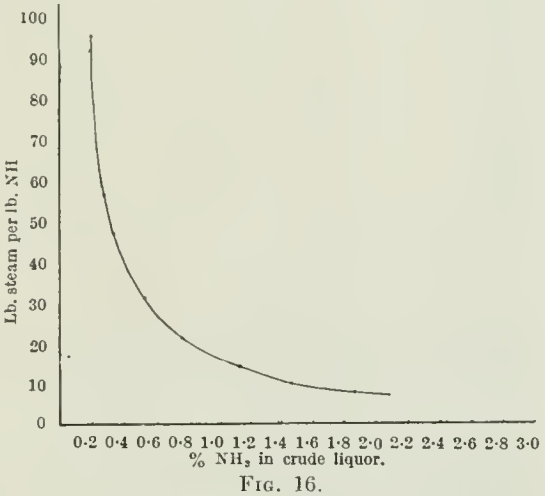


FIG. 16.

Curve showing steam consumption per pound of ammonia, with varying concentrations of ammonia in gas liquor.

by varying concentrations of ammonia in the gas liquor. This obviously has an important bearing when fixing the price of gas liquor according to its ammonia content, and particularly so when purchasing concentrated gas liquor. As far as is known, the only recent work published on this aspect was an article appearing in *Chemical and Metallurgical Engineering* of February 15th last by Emil Piron, in which some of the fundamental laws governing the distillation of mixtures were discussed. This worker has shown the lines along which problems of this character can be studied, both experimentally and mathematically. Indeed

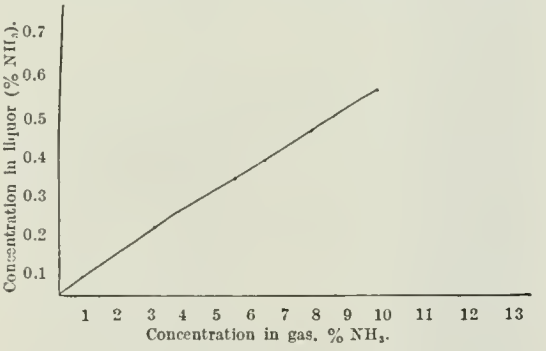


FIG. 17.

Diagram of Emil Piron's values, showing the variation of "K," with a variation of the strength of liquor.

the article is of such merit that diagrams, based on the experimental data furnished, have been embodied in this contribution. Fig. 17 is a graph which shows the relationship between the composition of the liquor being distilled and that of the distillate leaving the still. The particular experiment comes under the category of a discontinuous distillation, but the data so given can be corre-

lated to the performance of a continuous still This graph in conjunction with Fig. 18, which is compiled from data by the same author, is of importance as indicating the economic limit which should be looked for as representing the point at

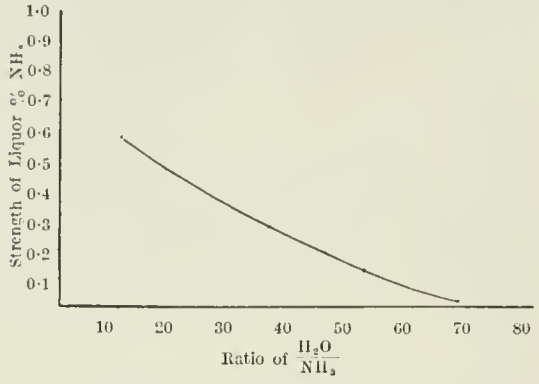


Fig. 18.

Steam consumption per unit of ammonia distilled from liquors of varying concentrations, plotted from data by Emil Piron (see Chem. and Met. Eng., Feb. 15, 1922).

which further distillation is unprofitable. It also furnishes some idea as to the relative value of gas liquor of different concentrations from the point of view of the distiller.

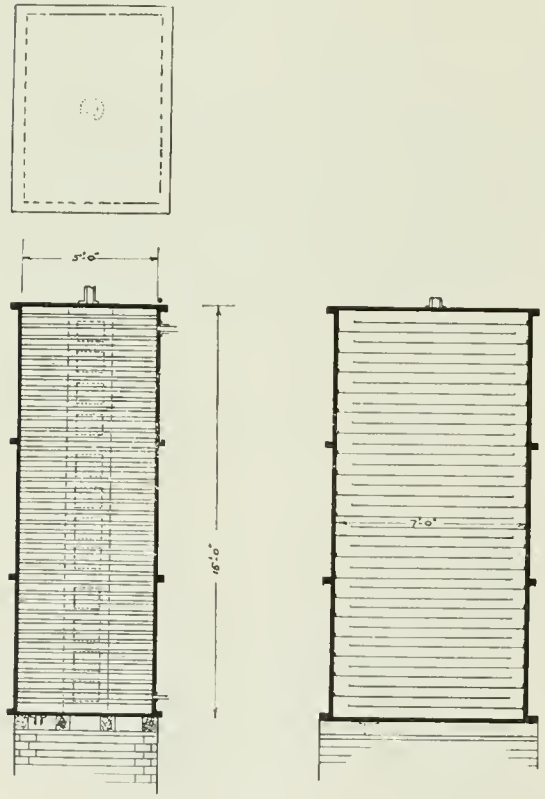


Fig. 19.

Hill's still.

It has been suspected by workers having technical and practical experience of gas liquor stills that there was no particular merit in the elements of the still being arranged with a deep seal, through which the ascending gases and steam were compelled to bubble. On the contrary, it has been suggested that the capacity of a gas liquor still can be fully maintained, and the steam consumption appreciably reduced, if the ascending gases and steam emerged on the liquor line in the trays.

Figure 19 shows a continuous gas liquor still which was in operation in London in 1878, the design of which was based on the principle of a comparatively large surface area with a thin film of liquor, along which the gases and steam skirted by reason of the contiguity of the respective plates. It is known that a still 15 ft. high by 7 ft. x 5 ft. in section, having 38 plates, was capable of dealing with the free ammonia content of 10,000 galls. of gas liquor of 6—8 oz. strength per day.

It is clear from the experience afforded by the operation of the above still that heating surface is not alone the governing factor which should be aimed at in the design of gas liquor stills. Rather must attention be directed to ensuring the greatest intimacy of contact along with maximum time contact and heating surface. Attenuated bubbling of such a nature that maximum deformation and/or reformation of the bubbles is ensured, is the principal factor for promoting rapid equilibrium.

Experiments have been directed to the elucidation of the above view by varying the depth of seal for a given still design, and although the author is reluctant to draw premature conclusions, evidences are not wanting which support the theory advanced above.

Little has been heard during the last few years concerning the distillation of gas liquor under vacuum. This question, however, was a "live one" more than twenty years ago when several stills were erected in Scotland for the distillation of ammoniacal liquor in the manufacture of ammonium sulphate under vacuum conditions. An interesting paper having reference to this subject was read before the Institution of Gas Engineers

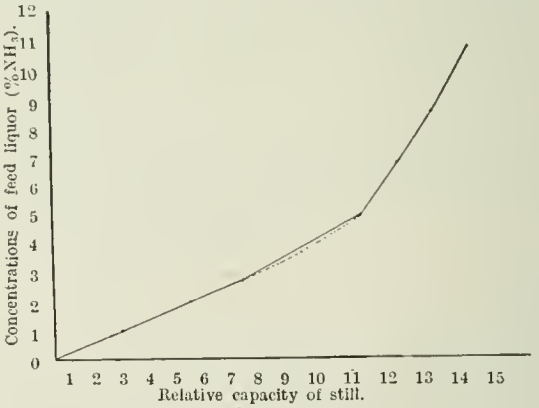


Fig. 20.

Diagram of the capacity of experimental still in relation to strength of feed liquor.

on June 11th, 1903, by the late Mr. Ballantyne, Engineer and Manager of the Hamilton Corporation Gas Department. Whilst the paper to a large extent was of a descriptive character, and did not afford much information of a positive technical nature, the view was expressed that the vacuum system of distillation was economical as regards steam consumption, particularly at those works where exhaust steam was available.

It has to be remembered that the utilisation of exhaust steam is not confined to vacuum distillation plants alone. Many gas liquor stills operating under normal pressure conditions are worked

to-day with exhaust steam. Moreover a consideration of the relative vapour pressure of ammonia and water in typical gas liquors at ranges of pressures between (say) 20 in. of mercury (absolute) and 35 in. (absolute) leads one to the conclusion that no great saving in steam consumption is likely to accrue, whereas an additional expenditure of steam will be required for the operation of the vacuum pump and the pump installed to withdraw the effluent liquor from the still.

Other considerations also operate when working a vacuum still. Some are advantageous, others have the contrary effect. The volume occupied by the steam at (say) 5·7 lb. per square inch (the absolute pressure at which the vacuum still at Hamilton is reported to have worked) is 68 cub. ft. per lb., as contrasted with 24·75 cub. ft. per lb. when operating under normal pressure distillation conditions. Thus, under vacuum conditions, the velocity of the steam through the still is appreciably increased and hence the time contact is reduced correspondingly for a given weight of steam. On the other hand, the greater volume of steam passing over, or bubbling through, the liquor on the several trays is clearly an advantage. Manifestly the elements constituting the chambers of a still operated under vacuum must have larger

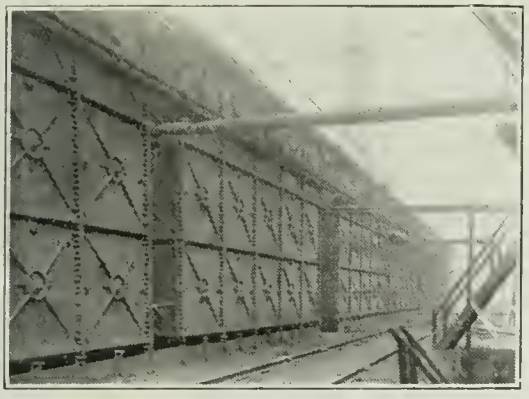


Fig. 21.

Photograph of gas liquor tanks.

surface area provided for the ascent of the stream of steam and ammonia.

As uncertainty existed as to the intrinsic capacity of a still for dealing with gas liquor of varying concentrations, experiments were made to determine this aspect. Gas liquor of varying concentrations was fed to the experimental still under such conditions that the effluent liquor leaving the still had approximately a constant composition. From the rates of flow and the concentration, Fig. 20 has been constructed. In brief, this curve represents the amount of ammonia distilled in unit time expressed in relation to the concentration of the feed liquor.

Having dealt in some detail with several aspects and considerations arising out of the design of ammoniacal stills, attention will now be directed to the essential parts of a unit for continuous distillation.

(*a*) Gas liquor storage tanks.
(*b*) Preheaters and coolers.
(*c*) Devil liquor storage tank.
(*d*) Constituent parts of gas liquor stills.
(*e*) Liming chambers.
(*f*) Seal pots.

Gas liquor storage tanks.

As to the form of the tanks and the general arrangement for the storage of gas liquor, practice varies considerably throughout the country. The shells of Lancashire boilers are used in many cases for the storage of gas liquor, and it is known that they have served quite well for this purpose.

It is usual at small and medium sized works to elevate the boilers on pier walls or even on the walls of the still house itself, so that the liquor to be fed to the still can gravitate to a constant feed tank provided with suitable float or special ball valve for ensuring a uniform feed of liquor to the stills. Experience suggests, however, that sectional plated cast-iron tanks are preferable, and are calculated to have a much longer life than second-hand steel boiler shells.

A typical installation of cast-iron tanks for a works distilling tolerably large quantities of gas liquor per day is shown in Fig. 21. Two such tanks are usually allocated to one unit. One tank in connexion with each unit contains the gas liquor for direct supply to the stills; the other tank being available for filling in the interim. Thus the tanks can be used alternately, and generally are of such a capacity as to afford twelve hours' supply. Each tank is approximately 25ft. x 15ft. x 15ft. The side plates are flanged and are 5ft. square by $\frac{3}{4}$ in. thick, and are arranged with centrally placed circular stiffening ribs from which diagonal stiffening arms radiate. The flanges are $\frac{3}{8}$in. thick, and are provided with stiffening webs. All flanges are machine finished. The joints are smeared with red and white lead ground thoroughly, embedded in which are lengths of twine. Two tie-rods 1$\frac{1}{2}$in. in diameter pass through each of the first two tiers of side plates, suitable bosses being cast on the horizontal centre line to receive these tie bolts. The top tier of plates are braced by a single 1$\frac{1}{2}$in. diameter tie bolt placed in the centre of the plate. The bottom plates are of the same size as the side plates, but are provided with a stiffening web in the centre running parallel with the flanges. Thus the flanges and stiffening ribs are arranged at about 2ft. 6in. centres. The bottom of the cast iron storage tank is supported by R.S.J.'s placed under the flanges and stiffening ribs at the centres just named. To prevent sagging of the tie-rods vertical angles are provided.

The outlet from the tanks is 6in. in diameter placed 18in. from the bottom of the tank. At the bottom of the tank a suitable pipe is fixed which is led to a separator for effecting the separation of any tar, which has settled out, from the gas liquor.

The tanks are provided at the top with deal joists and are covered with deal boards. From the top of the tank a 6in. diameter vent pipe leads to a small tower scrubber, which is packed with suitable filling and provided with an automatic water feed so that the vented ammoniacal air can be thoroughly washed and the ammonia arrested. The delivery pipe from the gas works liquor wells is led to within a few inches of the bottom of the tanks, so as to reduce the agitation of the gas liquor to a minimum.

The connexions from the storage tanks to the gas liquor pumps are made in cast iron pipe of the turned and bored type. The machining of these pipes is done to a definite gauge, and they are remarkable for their freedom from leakages.

The tanks are provided with floats which are connected by wire cord over suitable pulleys with a gauge board placed in the still house, by which the rate at which the liquor is being pumped to the stills can be accurately gauged and determined.

Preheaters and coolers.

A battery of preheaters and coolers in connexion with one of the units of the plant under review is shown in Fig. 22. The function of the preheaters is two-fold. Advantage is taken of the heat of the

waste gases and steam leaving the saturator to preheat the ammoniacal liquor prior to entering the still. In this way cooling of the waste gases is partially effected by the gas liquor, the final cooling being completed with water.

With small units of plant it is usual to place the preheater inside the still house, where it is not subjected to extremes of temperature during operation. With larger units of plant the preheaters are almost invariably placed outside the still house building. This practice may be regarded as unsound from the point of view of heat conservation. The quantity of heat leaving the saturator, however, is appreciably greater than that required to raise the temperature of the feed liquor to boiling point. Indeed, cooling water has to be profusely used to reduce the waste gases at atmospheric temperature.

The thermal aspect of this section of the plant will be the subject of further reference, as obviously this is one of the weak points in connection with current practice.

This type of heater will serve admirably for gas liquor free from tar, but it is suspected that if the gas liquor is of a dirty character the inside of the tubular undulations may become coated with tar, necessitating the dissembling of the sections. One distinctive feature which this type of heater possesses is the facility and comparative ease with which the segments can be dissembled either for cleaning purposes, or for renewal, in the event of fractures occurring.

Heater B is of a tubular form, and is arranged to take 61 cast iron tubes, each 3in. internal diameter by 3⅜in. external diameter. These tubes are provided with shoulders top and bottom, and each fits into suitable recesses in the corresponding tube plates, the annular spaces thus formed being caulked tight with a rust-jointing. To counteract the irregularities of expansion and contraction, as between the shell which externally is exposed to atmospheric influences and the tubes which are in contact with the hot waste gases, a suitable expansion joint ⊃ shaped, made of 32lb. lead is provided between the top flange of the heater body and the tube plate. This arrangement has been found effective in obviating fractures which at one time were experienced with this form of apparatus. The cast iron tubes constituting the heater must be cast without chaplets and must be tested to 40lb. hydraulic pressure.

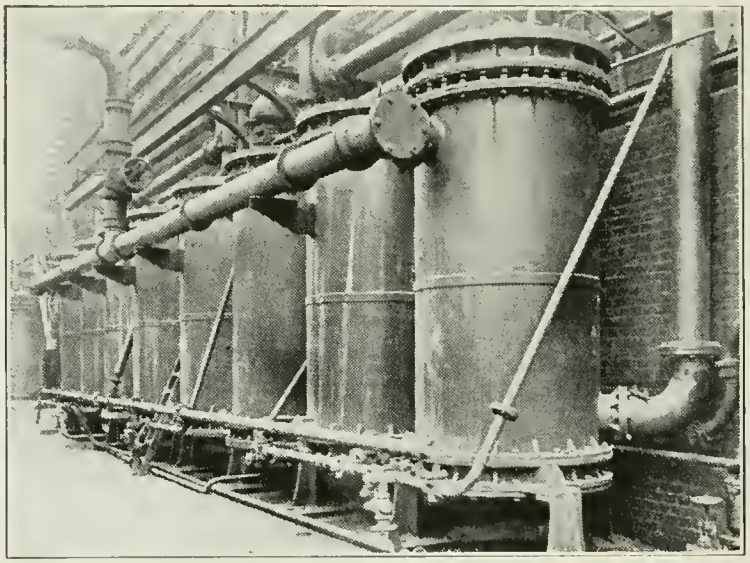


FIG. 22.

Photograph of pre-heaters and coolers.

Many forms of preheaters are used throughout the United Kingdom, dependent upon the size of plant for which they are intended and on the special experience of the maker.

Figures 23, 24, and 25 show three typical preheaters, (A), (B) and (C), each of which possesses distinguishing features.

Heater (A) consists of a series of double flanged cast-iron segments, alternate ones of which are void, and are provided with flanged inlet and outlet passages, the others being enclosed castings having a series of tubular undulations. The void chambers are coupled together by a series of flanged syphon bends which are placed left-hand and right-hand alternately. The liquor to be preheated enters at *a*, and is discharged to the still at *b*. The hot gases from the saturator enter at *c*, and leave at *d*. Manholes are provided in connexion with each of the enclosed chambers, so as to afford access for examination if required. One 4ft. diameter heater as shown has an effective surface of 634 sq. ft., and is capable of preheating ninety tons of 8 oz. gas liquor per diem. This represents 7.04 sq. feet per ton of gas liquor per diem.

It has been found in practice that four vessels of the type represented by B are necessary for a still dealing with 30,000 gallons of liquor per day if the gases are to be cooled sufficiently and to be deprived of water vapour to such an extent as to allow of combustion in spent oxide burners. Under such conditions two vessels serve as heaters and two as coolers. The gas liquor is fed to the bottom of the second heater in series and leaves at the top, passing again to the bottom of the first heater and leaving at the top to the still. It is important that the liquor should be fed to the bottom in all cases so as to obviate trouble from air locks.

When the vessels serve as coolers it has been found advantageous to insert a circular-drilled cast-iron plate with 61 1in. to ¼in. taper holes so as to ensure uniform distribution of the cooling

water. Each heater has an effective surface area of 464 sq. ft. By calculation it will be seen that this represents 6.18 sq. ft. of heating surface per ton of gas liquor per diem.

Heater *c*, which is rectangular in section, has obviously been designed to facilitate replacement of any of the tubes which may fracture or develop leakages without obviating a stoppage of the heater. It consists of seven separate elements, four being placed on one side and three on the other. The waste gases from the saturator enter at *c* and leave at *d*. The gas liquor to be preheated enters at seven different points coinciding with A. This heater affords an effective surface area of 144 sq. ft., and is capable of preheating 14½ tons of 8-oz. liquor, representing 8 sq. ft. per ton of liquor per diem. It is not improbable that this heater is capable of dealing with a larger quantity of liquor than that indicated, as considerable advantage must accrue by reason of the tubes being placed horizontal to the flow of the gases. Such an arrangement admits of a turbulency of motion which is singularly absent in many types of multi-tubular heaters.

All the preheaters previously described have suitable provision for automatically running the liquid which condenses from the waste gases, and which on account of its disagreeable smell and character has been appositely called "devil liquor." The devil liquor outlets from the preheaters and coolers are suitably sealed to prevent the escape of the waste gases which consist of hydrocyanic acid, hydrogen sulphide, and carbon dioxide, etc.

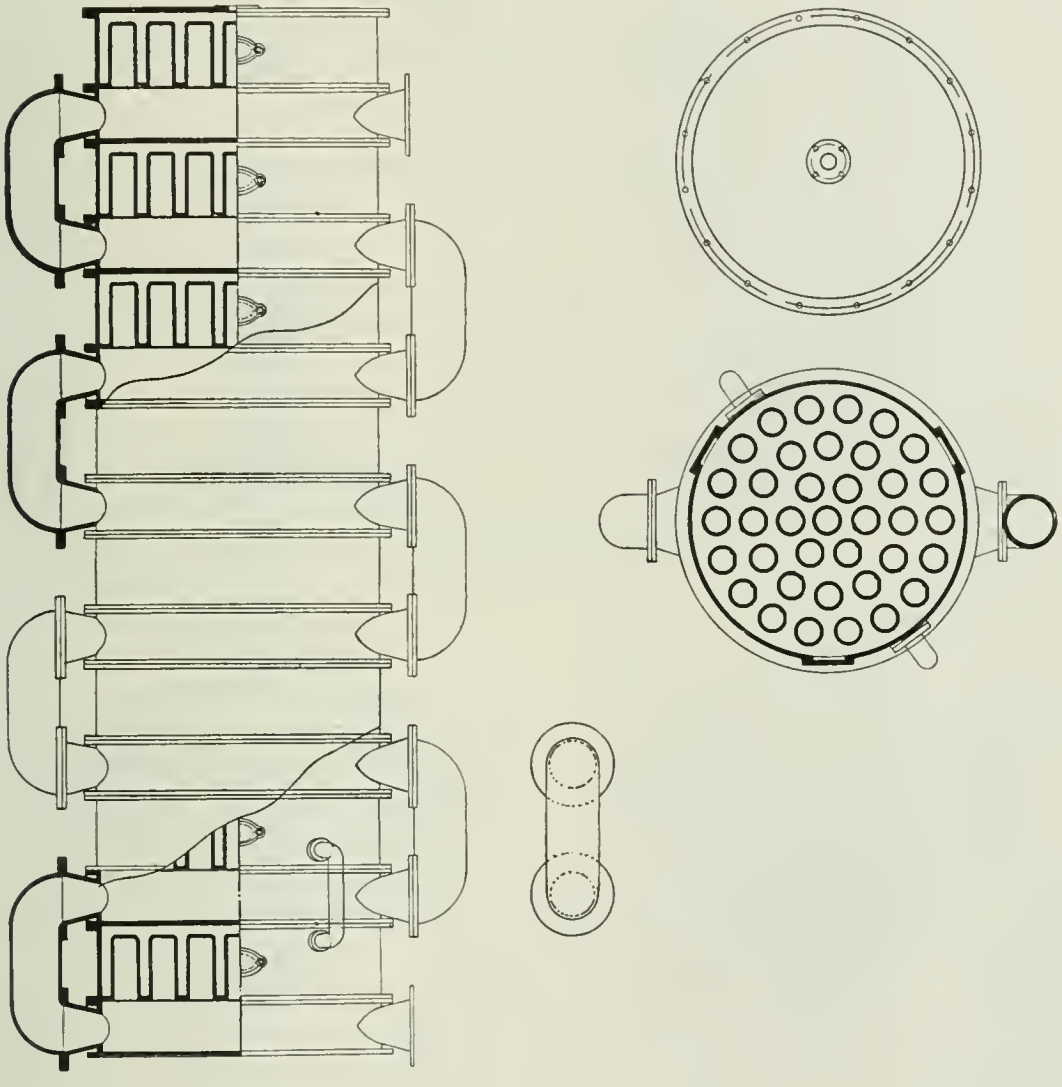


FIG. 23.

Heaters.

For small and medium sized works, preheaters consisting of two circular double-flanged pieces, with a deeper double-flanged piece for the body, have been fairly extensively adopted. Two plates are placed between the top and bottom flanges of the body and the double flanged pieces. Wrought iron tubes are used and are expanded into these tube plates.

The author's experience of the use of wrought iron tubes has not been too satisfactory owing to

corrosion arising at the cooler parts of the tube due to the formation of iron sulphide and the influence of the hydrocyanic acid of the waste gases.

Of course it is recognised that a better transmission of heat is possible owing to the thinner walls of the wrought iron tubes as contrasted with the cast iron ones, and equally it is appreciated that the greater resiliency and tensile strength of wrought iron tubes are decided factors in their favour. Notwithstanding these advantages, the author does not hesitate to recommend cast iron multi-tubular preheaters.

The life of any preheater, whether provided with cast iron pipes or wrought iron pipes, is materially influenced by the freedom with which the waste gases and steam are deprived of vesicular ammonium sulphate containing acid, which is mechanically carried forward from the saturator. Cast or wrought lead baffle boxes of a suitable form must be provided, and during operation of the plant steps must be taken to ensure that they operate efficiently at all times.

Devil liquor storage tank.

On account of the noxious character of the devil liquor, it is important that certain steps should be taken to ensure its collection and treatment. This is best effected in a sectional plated cast iron tank suitably lagged which will allow of 5 to 6 hours' accumulation. Opinions vary as to the best method of treatment. At many works the devil liquor is pumped along with the gas liquor and the mixed liquors are distilled. The only disadvantage is that expense is entailed in the matter of steam, and many of the impurities of the gas liquor which are arrested with the devil liquor, such as pyridine, creosote oil, naphthalene, etc., are again returned to the saturator. Obviously for the production of white salt it is essential that these impurities should be disposed of in another way, and not recirculated to the saturator.

Moreover, it must be remembered that the distillation of devil liquor represents approximately a 20% reduction of the capacity of the still, which is another important consideration.

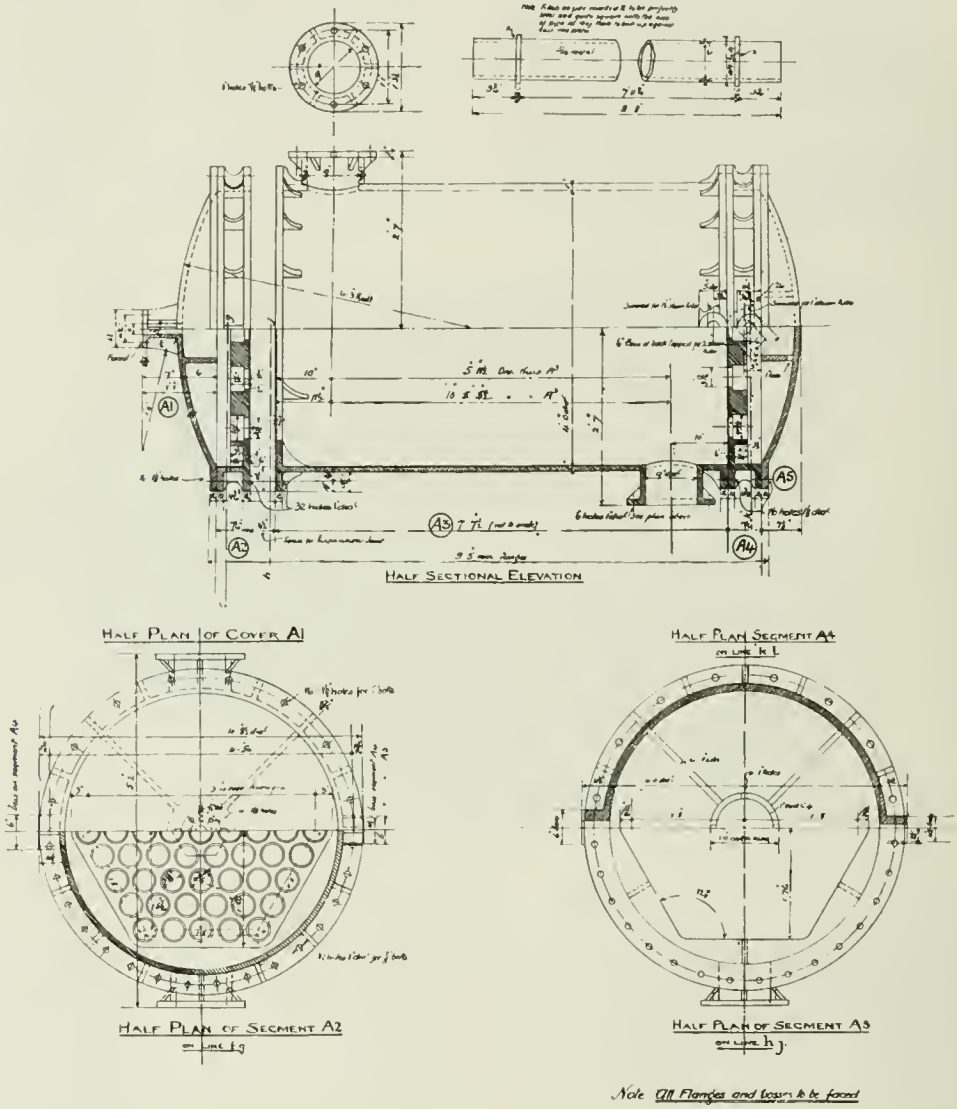

HALF SECTIONAL ELEVATION
HALF PLAN OF COVER A1
HALF PLAN SEGMENT A4
HALF PLAN OF SEGMENT A2
HALF PLAN OF SEGMENT A3
Note All Flanges and Lugs to be faced

FIG. 24.
Heaters.

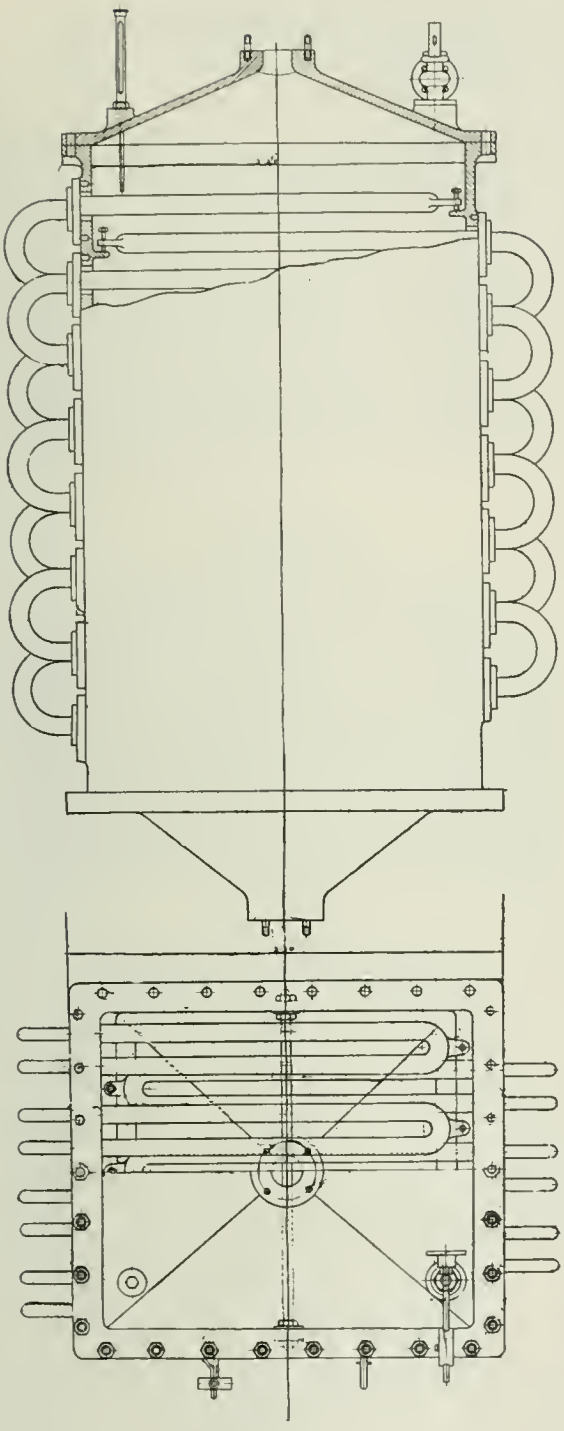
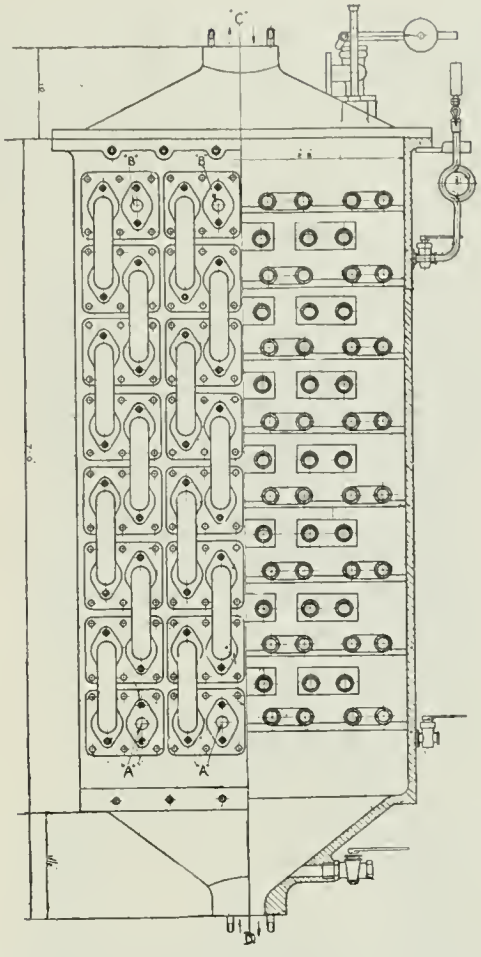


FIG. 25.
Heaters.



NOTE.—When heater is used for heating ammoniacal liquor, the liquor enters at "A" and leaves at "B." The hot foul gases from the saturator enter the heater at "C" and leave at "D."

Constituent parts of gas liquor stills.

Figure 26 shows how the elements of the still illustrated in Fig. 5 are assembled. The bottom tray is supported on a circular webbed flange arranged within the still. Each succeeding tray is in turn supported by the one underneath. The first bubbler of the free still, placed immediately above the liming chamber, is supported by a vertical 9in. cast iron pipe (which by a bend near the foot conducts the limed liquor to an outer annulus) placed centrally in the liming chamber and terminating with a suitable base. The first bubbler supports the hood which in turn supports the bubbler. Thus the whole weight of the bubblers and hoods is transmitted to the 9-in. pipe, and the complete weight of the trays is reflected on the webbed flange.

For the permanent machined joints a mixture of red and white lead is used. For the faced joints of the superimposed sections graphite paste is used. A packing of yarn is also employed between the trays and the outside shell of the still so as to prevent the admission of any corrosion which will interfere with the removal of the trays. It should be remarked that the bubblers of the fixed section of this particular still are open, with serrated edges on the two circumferential peripheries.

The merits of the design of this still are the size of the liquor overflows and the large area of wetted surface which the elements afford. Further, it is evident that the designer has appreciated two other important factors, viz., the great advantage of a high velocity of the vapours where they come in contact with the liquid, and the importance of time contact as between the vapour

above the liquid and the liquid itself as aiding equilibrium.

Figure 27 shows the elements of a still which was introduced to this country about 40 years ago. Some of the features of this still, particularly the liming section, possess definite merit, but it is believed that there are many directions in which the still can be improved, particularly in the matter of area of liquor overflows, time contact between the vapour and liquid, and velocity of the vapours through the liquid.

Figure 28 represents the elements of a still of more recent design. Regard has obviously been paid

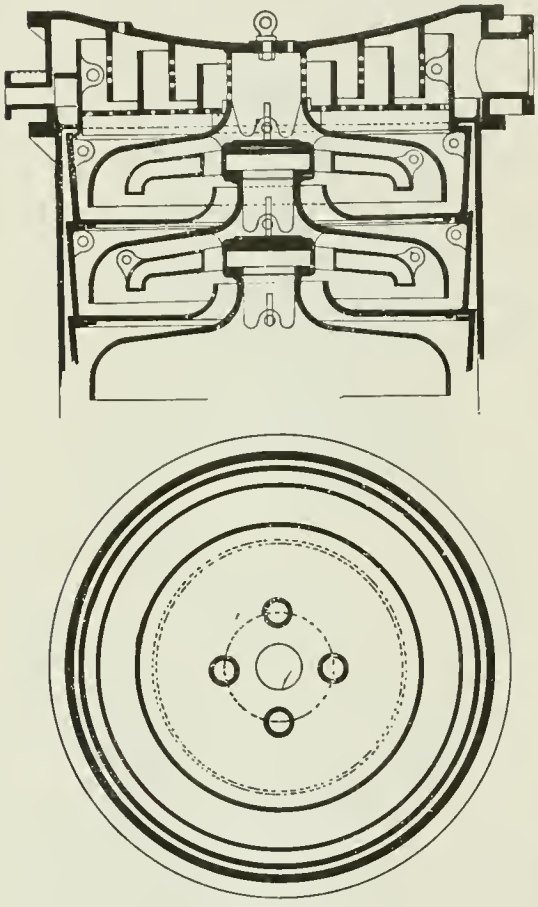


Fig. 26.
Elements of Gas Liquor Stills.

to the removal of the hoods for cleaning purposes, and the possibility of blocked outlets would appear to be remote, in view of their size. The comparatively small diameter of the still in relation to the quantity of gas liquor with which it deals and the arrangement of hoods which clearly admit of the bubbles from one being thrown in contact with those created by the adjacent hood, must ensure considerable deformation, thus increasing the contact of liquid and vapour.

Figure 29 represents a very simple form of element. If simplicity were the predominant factor in the efficiency of a gas liquor still, this particular design would certainly merit a premier place. The hoods in both the free and fixed stills are rectangular and the travel of the liquid is left to right and right to left alternately. The overflows are tolerably large and the designer has evidently had in mind the importance of avoiding

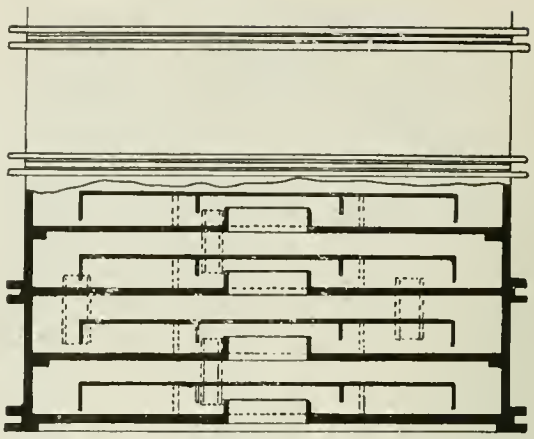
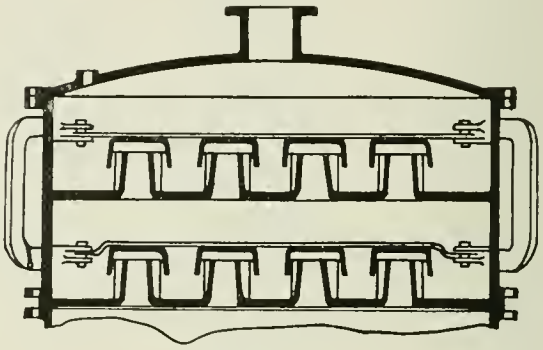


Fig. 27.
Elements of Gas Liquor Stills.



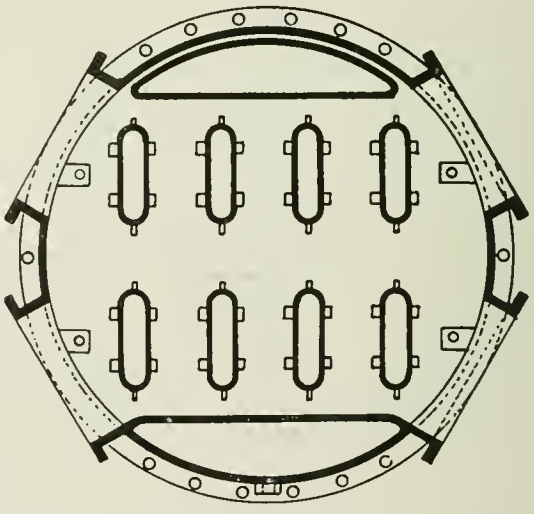


Fig. 28.
Elements of Gas Liquor Stills.

blockages at this point. The hoods are capable of ready removal. One disadvantage appears to be that the arrangement of hood lends itself to localised bubbling. Moreover the area of the serrations is equal to, if not in excess of, the area of the gas slots, which area is too large for ensuring such a vapour velocity as to give anything approaching optimum bubbling.

Figure 30 represents the elements of a square still which by reason of its form must have the merit of cheapness as regards cost of production. The bubbling hoods are also readily accessible either for inspection or withdrawal. Whether the liquid will follow the path intended for it appears somewhat open to doubt. Whilst the arrangement of four hoods is a considerable advantage, it is feared that the area for the ascent of the gas is far too large for ensuring satisfactory bubbling, and the liquor overflows also appear to be unnecessarily restricted, although protruding as they do beyond the castings, they are without doubt easy of access.

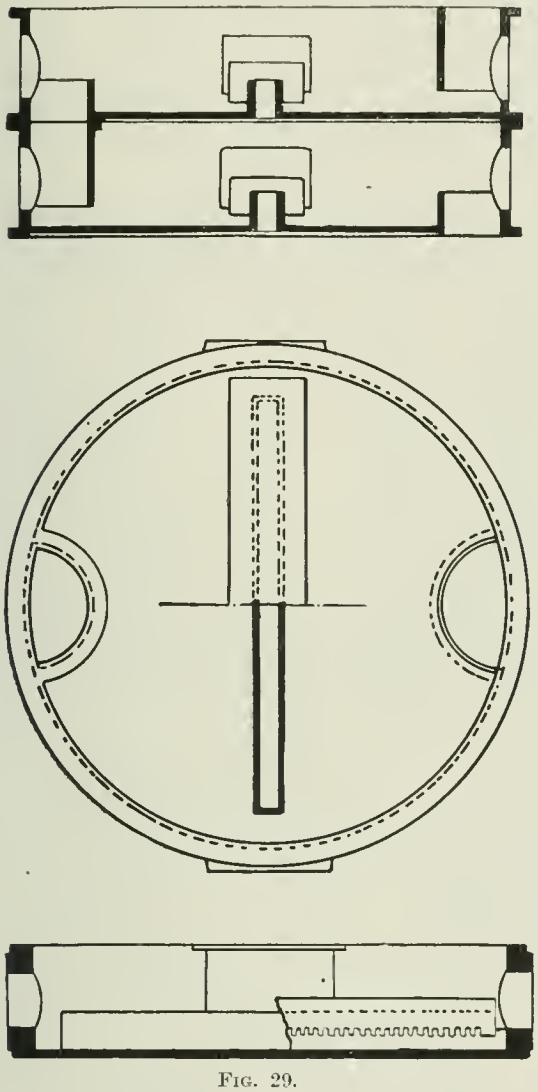


FIG. 29.
Elements of Gas Liquor Stills.

Figure 31 represents the constituent parts of a still of recent design. This still has a very large capacity. The designers have recognised the importance of large overflows and the advantage attending the depth of chamber in relation to the liquor on the trays as increasing the factor of time contact. Moreover the weight of the still in relation to the capacity is certainly a chemical engineering achievement. The latter is largely due to the adoption of a small liming chamber, and of course introduces a subject on which there would be, no doubt, considerable divergence of views. It is believed that the design, by reason of the large area afforded by the serrations, militates against such a vapour velocity as is calculated to ensure satisfactory bubbling.

Figure 32 represents the elements of a still possessing several merits, particularly the one of creating

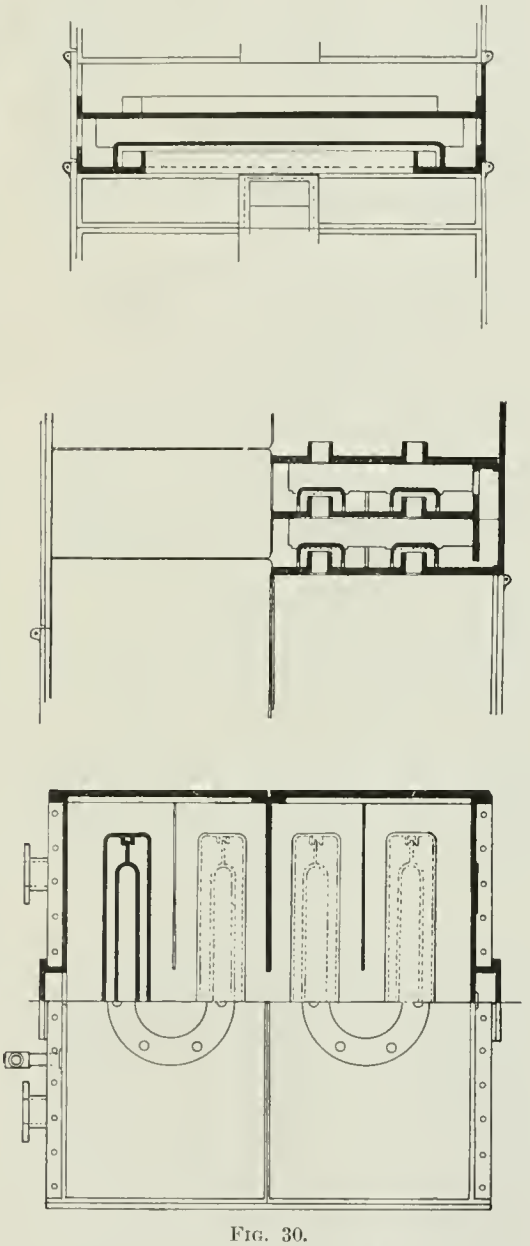


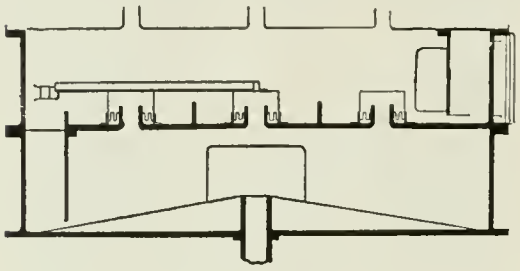
FIG. 30.
Elements of Gas Liquor Stills.

bubbling over a large surface area, and ensuring impact between the respective bubbles from the several hoods. It will be an advantage if the makers will in future increase the diameter of the overflow pipes, as it is not inconceivable that trouble may arise from this cause.

Figure 33 represents the chief constituent parts of a still which was designed nearly forty years ago. Whilst the designer has apparently recog-

nised the importance of maximum agitation of the liquid, it is feared that the area of the serrations, which are largely in excess of the area of the gas outlet, will obviate this desideratum.

Figure 34 represents the elements of a square still, certain advantages of which have already been



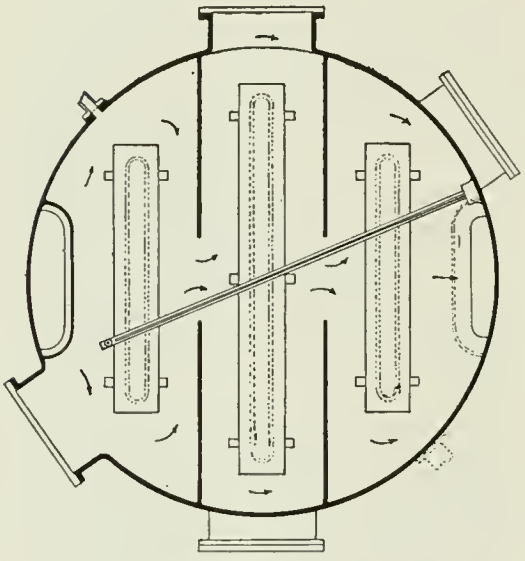


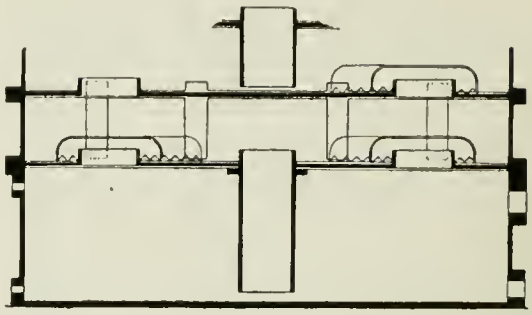
FIG. 31.
Elements of Gas Liquor Stills.

named. Here the importance of vapour velocity appears to have been recognised, although impact of the bubbles appears impossible. The liquor overflow might with advantage be enlarged.

Figure 35 represents the details of a still which it is understood is used to a considerable extent in connexion with ammoniacal liquor arising from the distillation of shale. The position in which the bubblers lie is varied alternately with each chamber. Thus they cross at right angles. Similarly the overflows are arranged alternately. This design, it is feared, does not allow of a sufficiently rapid vapour velocity to ensure satisfactory bubbling, although the arrangement of the hoods and their comparative contiguity will doubtless assist in causing conflict between the respective bubbles.

Figure 36 is similar in many respects to the elements of one of the other square stills to which reference has already been made.

Figure 37 represents the sections of a still which has obviously been designed to ensure agitation over a large area, but unfortunately the low velocity of the gas is not calculated to assist to this end. The manholes appear to afford satisfactory access to the overflow pipes, but whether



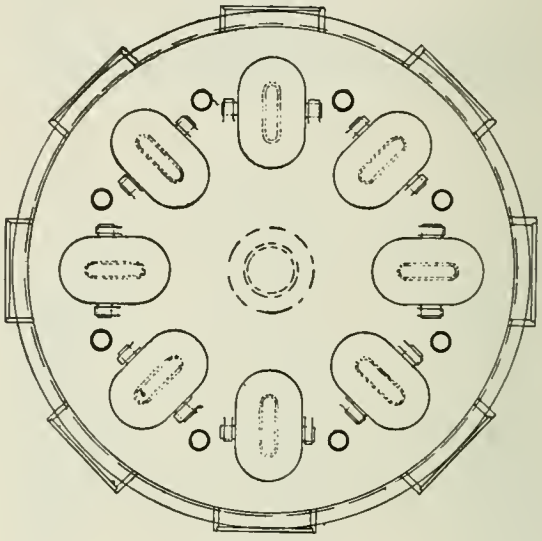
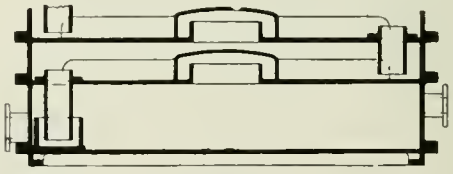


FIG. 32.
Elements of Gas Liquor Stills.



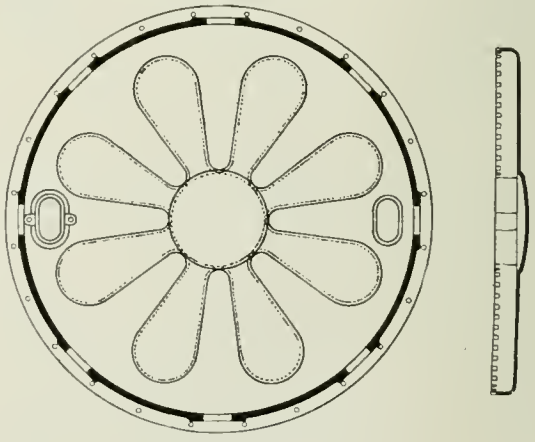


FIG. 33.
Elements of Gas Liquor Stills.

the serrations of the hoods can be as easily cleaned, or whether they can be readily withdrawn, is a matter of doubt. If there had been a larger

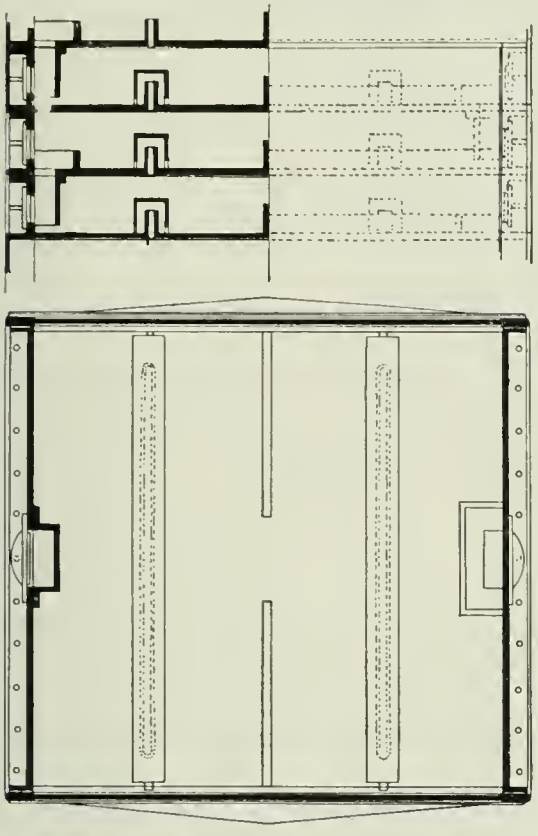


FIG. 34.
Elements of Gas Liquor Stills.

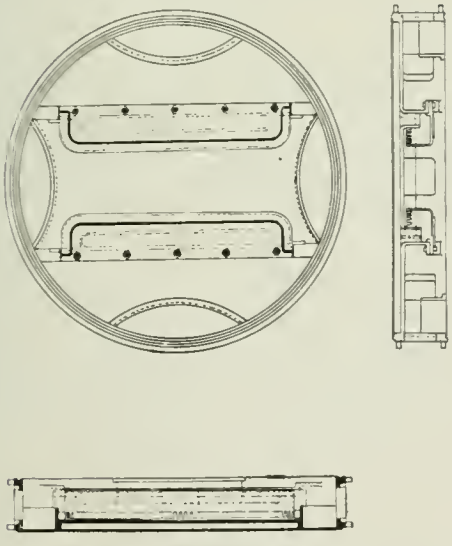


FIG. 35.
Elements of Gas Liquor Stills.

vapour space above the liquid level in the trays, this would have ensured a greater time contact which obviously is a decided advantage.

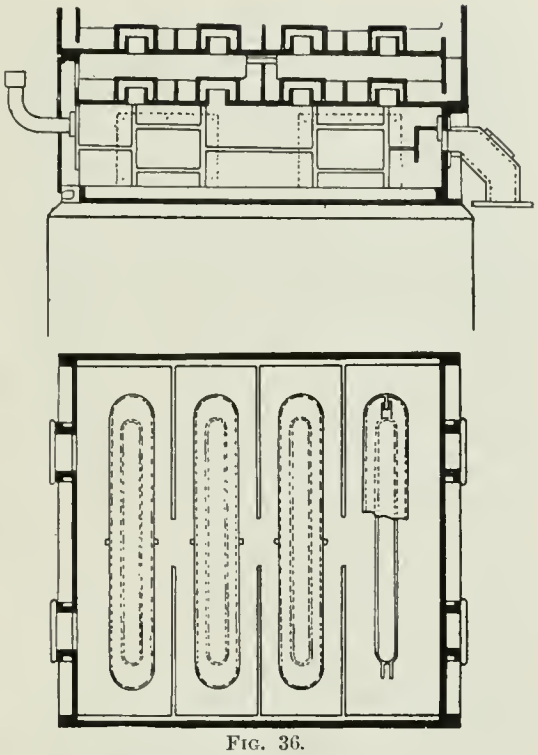
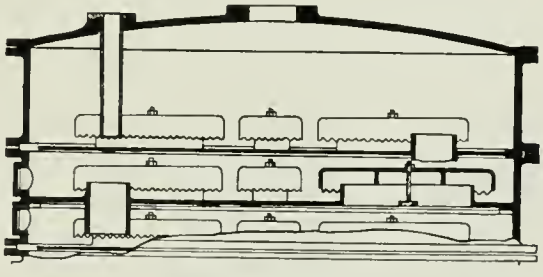


FIG. 36.
Elements of Gas Liquor Stills.



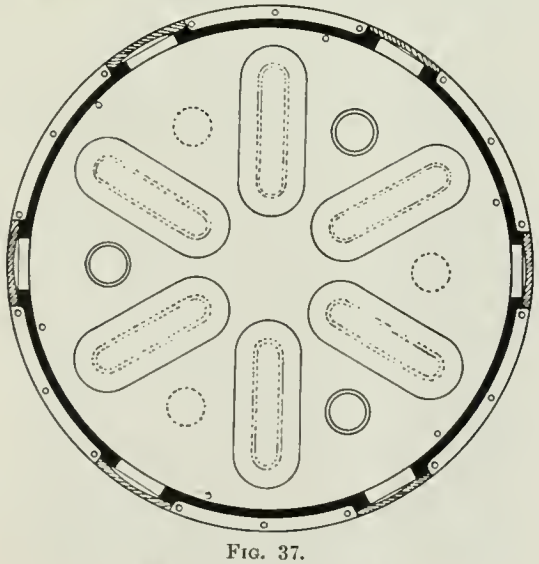


FIG. 37.
Elements of Gas Liquor Stills.

"Liming" and liming chambers.

The most economical way of liming the gas liquor, which has been deprived of its volatile ammonia, is to introduce powdered lime by a positive method to the liming chamber. By pursuing this method steam and water are economised, and in addition advantage can be taken of the heat generated by the formation of calcium hydroxide. The importance of this aspect is made evident by the graphs and data furnished in Fig. 19.

Unfortunately lime containing 96 per cent. CaO and free from ashes is very difficult to obtain, and no doubt this has been one of the deterring factors to the adoption of such a method as the one indicated. It is found in practice that cream of lime cannot be pumped satisfactorily, and that if pumping is resorted to milk of lime of about 16° Twaddell at 70°-75° C. has to be used. The lime mixer must obviously be placed in such a position that the cream of lime (say) of 30° Tw. can be gravitated direct to the liming chamber. When regard is had to the fact that lime is with difficulty soluble in water (only a 0.17 per cent. solution at 15° C. being possible) the importance of maintaining the cream of lime in agitation with the liquor need not be unduly emphasised.

The introduction of cream or milk of lime to the still chills the liquor at the foot of the liming chamber momentarily, but sufficient steam, usually about 10 per cent. of the total, has to be added to ensure the necessary agitation and to maintain the mixed liquor at boiling temperature.

Coming to the question of the size of a liming chamber, it would appear evident that if agitation is to be efficient, there must be a limit to the size of the liming chamber in relation to a given volume of limed-liquor treated. Undoubtedly one of the faults of design of many liming chambers is that they are too large and allow calcium salts to settle out, instead of being retained in a state of suspension. This is a matter to which increasing attention could be very profitably devoted.

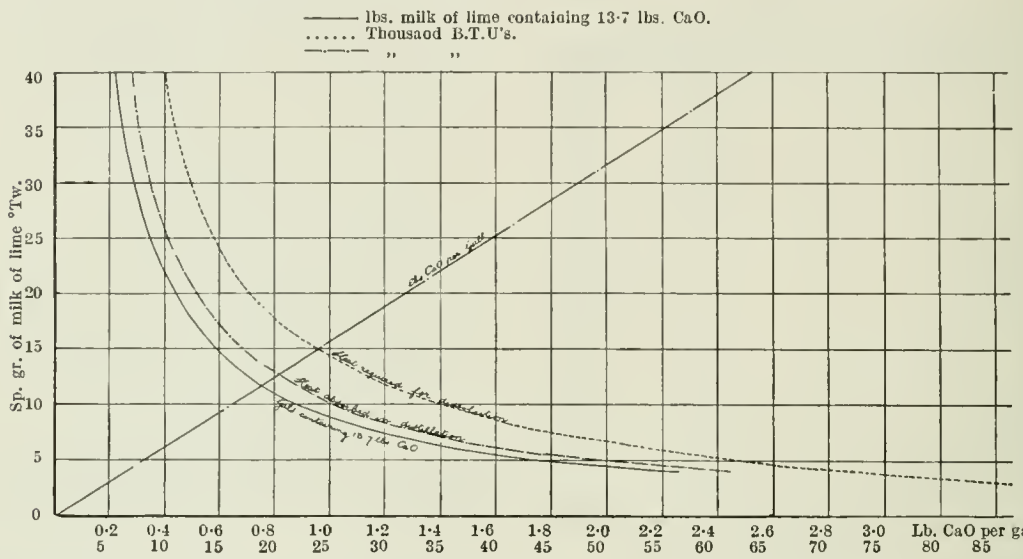
Not infrequently has the question been asked, by managers in charge of small and medium sized sulphate of ammonia plants, whether any formulæ were known relative to the quantity of milk of lime required per unit volume of gas liquor containing a certain percentage of fixed ammonia in relation to the total ammonia.

Figure 38 is of interest in this connection. It is clear that a formula of the kind referred to can be readily calculated from analytical data. One such formula (based on the milk of lime required per hundred gallons of gas liquor) of which the writer has knowledge is expressed thus:—

$$V = 65\ F \div L,$$

where V represents the volume of milk of lime in gallons, F the fixed ammonia content of the gas liquor in ounce strength, and L the strength of lime in degrees Twaddell.

The following calculation will afford an indication as to how the above formula has been arrived at, and incidentally gives an idea of the excess milk of lime solution which it contemplates:—



Sp. gr. (°Tw.)	4	8	12	16	18	20	22	24	26	28	30	32	34	38	40
Lb. CaO per gall.	0·244	0·498	0·752	1·00	1·13	1·26	1·38	1·52	1·64	1·77	1·90	2·03	2·16	2·42	2·55
Galls. required to give 13·7 lb. CaO	56·1	27·5	18·2	13·7	12·1	10·9	9·9	9·0	8·4	7·7	7·2	6·7	6·3	5·7	5·4
Lb. required to give 13·7 lb. CaO	572	286	193	148	132	120	110	101	95	88	83	78	74	67	65
A. Heat required for dissolution, B.Th.U. (1000's)	86·4	43·2	29·2	22·4	19·9	18·4	16·6	15·3	14·3	13·3	12·5	11·8	11·2	10·1	9·8
B. Heat absorbed in distillation, ditto	60·9	30·5	20·5	15·8	14·1	12·8	11·7	10·8	10·1	9·4	8·8	8·3	7·9	7·2	6·9

NOTES.—1. The above figures are calculated for the distillation of 100 galls. of 8-oz. liquor having a fixed ammonia content of 0·346% w/v (20% of the total ammonia).
2. An excess of 20% above theoretical lime quantity is allowed.
3. The milk of lime enters the still at 110° F. and has therefore to be raised through 106·5° to 216·5° (the temp. of distillation).
4. The heat required for the dissolution of the lime is taken as 151 B.Th.U. per lb. of solution.

FIG. 19.

Diagram having reference to the thermal aspect of the application of lime.

1 oz. sulphuric acid = 0.3469 oz. NH_3.
1° Tw. milk of lime = 0·845 % CaO at 70° C.;
or, in 100 oz. ditto there is 0·845 oz. CaO
in 1 gallon ditto ,, ,, 1·352 ,, ,,
2 NH_3 : CaO as 34 : 56

1 oz. fixed ammonia requires $\frac{0{\cdot}3469 \times 56}{34}$

$= 0{\cdot}5712$ oz. CaO.

The factor per gallon of gas liquor on the above given data is $0{\cdot}5712 \div 1{\cdot}352 = 0{\cdot}42$, or a factor per 100 gallons of 42.

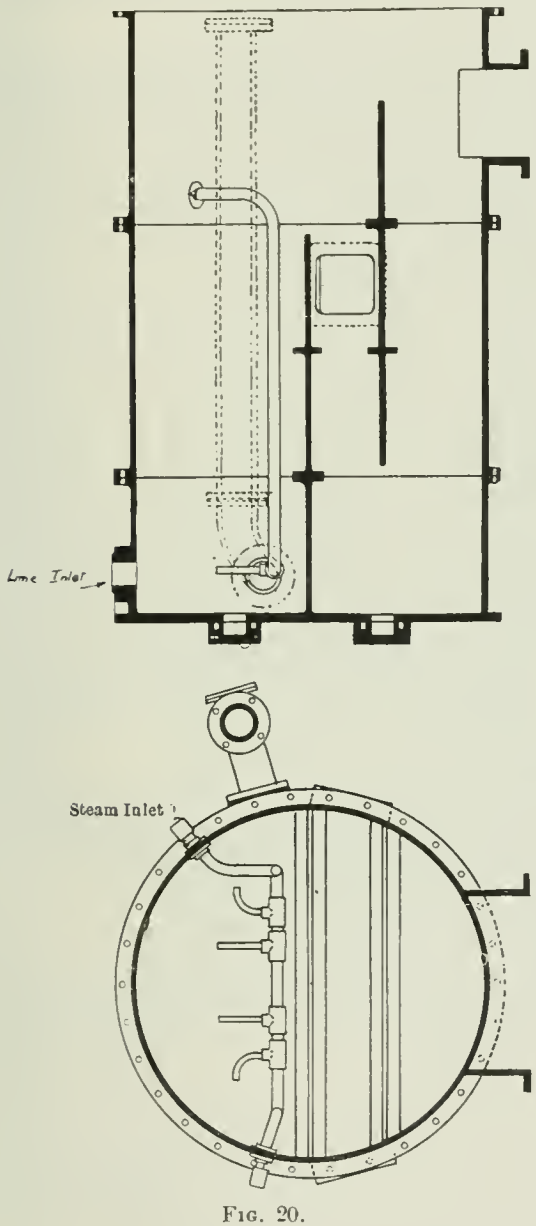


Fig. 20.
Liming Chambers.

Presumably the formula cited contemplates about a 50% excess of milk of lime. This is rather excessive as extended experience suggests that the whole of the fixed ammonia can be eliminated, given reasonable control with a surplus of milk of lime of 10—20% of the theoretical.

Coming now to the question of typical liming chambers, Fig. 20 shows a pipe bringing the gas liquor to be limed to the foot of the still, at which point the milk of lime is added. The steam inlet pipe is provided with four outlets two of which are placed tangentially, the other two being at right angles to the pipe. The baffle plates admit of considerable travel for ensuring uniformity of admixture.

Figure 40 represents another form of liming chamber. The liquor to be limed is brought to the foot of the still by four pipes and the milk of lime is admitted at the foot of the still, a suitable agitating steam pipe with radial arms being provided. The limed liquor ascends the liming chamber and is filtered through a perforated plate prior to being discharged to the fixed still. Whether blockages arise on the perforated plate is not known, but except the agitation is tolerably effective, it is feared that this may prove a possible source of difficulty, particularly so as the perforated plate is not too readily accessible.

Figure 41 shows a simple form of liming chamber

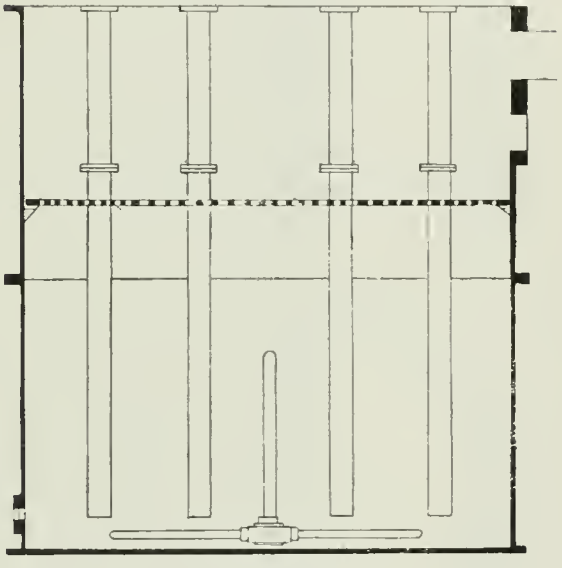


Fig. 40.
Liming Chambers.

which intervenes between the free and fixed stills. The liquor to be limed enters almost at the foot of the chamber, at which point the milk of lime is introduced. Agitation is created by a circular perforated pipe, the limed liquor leaving by the overflow pipe shown in the sectional elevation.

Figure 42 represents another typical liming chamber having many features in common with those already described. It will be seen that this arrangement provides for the limed liquor passing to a chamber at the foot of the column before being led to the fixed still.

It is important that the gas liquor should be thoroughly decarbonated before entering the liming chamber, otherwise the ammonium carbonate will react with the milk of lime forming calcium carbonate, which becomes hard in the still and renders cleaning much more frequent and difficult.

Where the liming chambers are of tolerable size, provision should always be made for the periodic emptying of the liming chamber in order to remove the accumulations of lime sludge which settle out.

Indeed, it has been found advantageous in the operation of many stills to run off part of the limed liquor from the liming chamber direct to the waste liquor main once or twice each day.

Manholes should also be provided in connexion with all liming chambers in order to afford access for cleaning purposes.

Seal pots.

To prevent the escape of steam etc. from the foot of the fixed still or fixed segment, it is necessary to provide some suitable seal pot or equivalent arrangement in order to ensure that the steam admitted to the fixed still will proceed through the several chambers and not by-pass the apparatus. Seal pots usually take the form of a double-flanged pipe with blank plates, top and bottom. The waste liquor pipe is provided with a regulating cock immediately on the outlet from the fixed still, and this pipe is led in some cases to the foot of the seal pot which is provided with an outlet near the top, or, the waste liquor pipe is fixed inside the seal pot, discharging the waste liquor near the foot, the outlet from the pot being near the top. The former type is placed on the floor level: the latter type is sunk into the ground. It is preferable that a vent pipe should be provided in connection with the seal pot so as to prevent steam "locks."

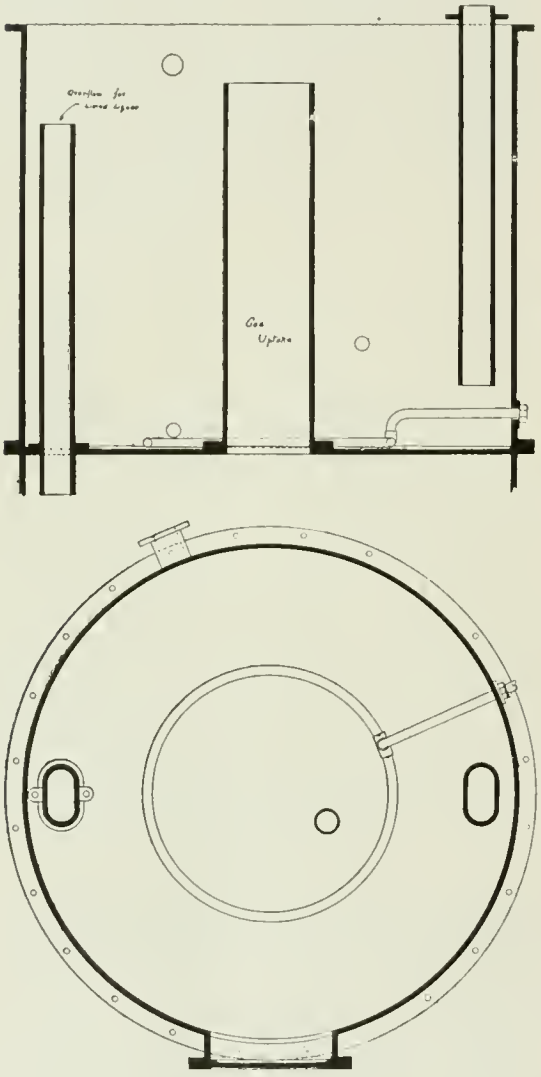


FIG. 41.

Liming Chambers.

An adjustable type of sealing device which is quite a commendable arrangement is shown in Fig. 43.

There are three essential factors affecting the efficiency and success of any continuous distillation operation, viz., constant steam supply, constant feed and constant discharge.

Constant steam supply.

The pressure against which the steam has to work in the fixed still, where usually 90% of the total steam is admitted, is the pressure of the combined seals in the several trays, plus the seal of the cracker pipes of the saturator, and the skin friction of the preheaters, coolers and pipe lines. This pressure will obviously vary with different stills, but usually does not exceed 3½ lb. per sq. in.

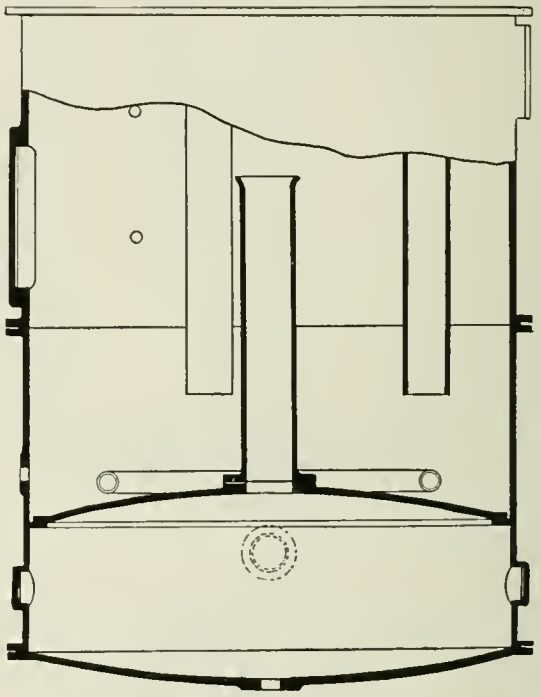


FIG. 42.

Liming Chambers.

In the case of the steam admitted to the liming chamber, the pressure against which this will have to work will be of the order of 5—6 lb. per sq. in. It is clear, therefore, that some form of reducing valve must be used, in order to reduce the pressure at which the steam boilers normally work to a pressure compatible with the requirements of the stills.

Figure 44 shows the reducing value with constituent parts, which is found to be effective for the purpose in question. The high-pressure steam enters tangentially and passes through the slotted holes of the cage, and the extent to which the spindle, which carries two inverted mushroom-type valves, is weighted, determines the size of

the apertures, through which the steam has to pass, and incidentally governs its pressure. Suitable drainage provision for any condensation is made.

Constant feed of liquor.

Many arrangements for ensuring a constant feed of liquor to ammoniacal liquor stills have been devised. Generally speaking, those having a float or suitable steel ball valve placed inside a small tank are the ones in operation in connexion with small and moderate sized distillation units.

An arrangement for ensuring satisfactory and uniform feed in connexion with large units of plant is shown in diagram Fig. 45. This arrangement provides for direct pumping of the liquor to the still, the rate of feed being governed by a cock to which is fitted a graduated quadrant and pointer. As a means of governing the delivery of liquor from the pump a connection is made from the delivery line to the suction, and inserted in this line is a spring-type of by-pass valve, the details of which are illustrated in Fig. 46. The principle of this by-pass valve is that the

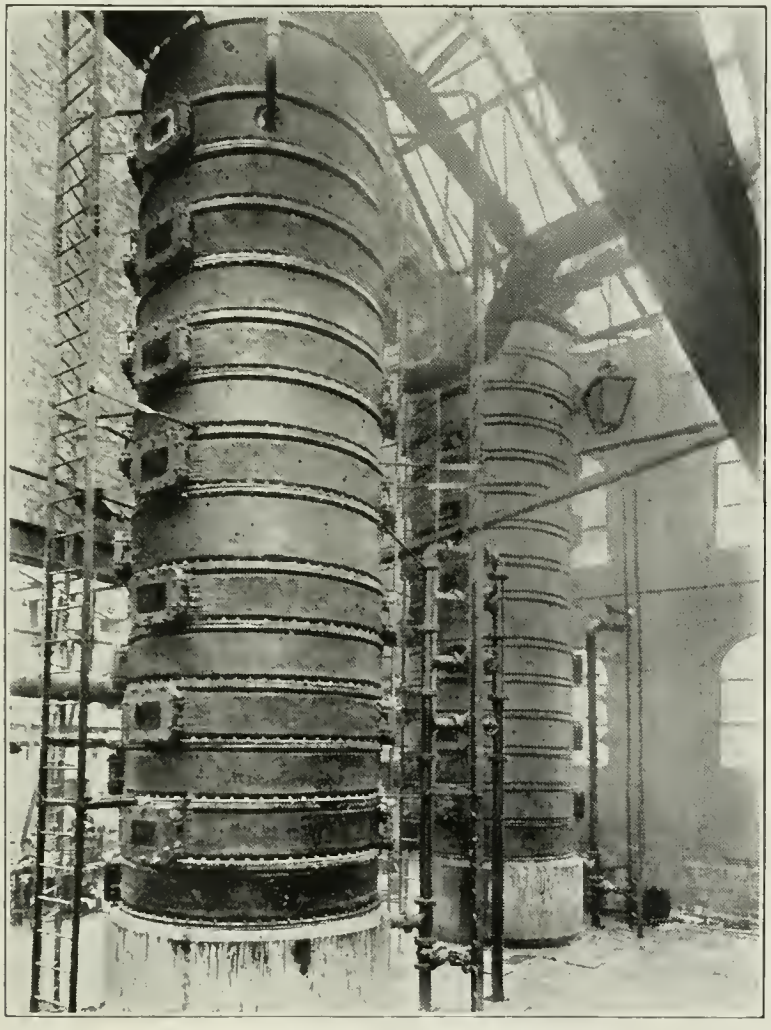


FIG. 43.

Photograph showing an adjustable seal arrangement in connection with gas liquor stills.

On the whole this type of constant feed arrangement has been found fairly satisfactory in operation. Some makers provide a graduated sight feed arrangement, consisting of a globe inside which is placed a pipe having a series of vertical stepped holes, graduated to represent varying rates of flow.

The only disadvantage of either of the above arrangements is the fact that the pressure against which the liquor is discharged is a slightly varying one, dependent on the depth of the acid seal in the saturator.

liquor floats the mushroom valve against the pressure of the spring which can be adjusted.

Constant discharge.

This is dependent almost wholly on the maintenance of the two conditions to which reference has just been made. It has been found in practice that the setting of the outlet cock can best be adjusted by providing it with a graduated quadrant and pointer. In starting a new unit, it will be assumed that the erectors have definitely ensured that the trays are dead level, other-

wise a grave defect, prejudicially affecting the efficiency and subsequent working of the plant has occurred.

It is known that unsuspected mal-alignment of the elements of stills has been the cause of considerable trouble in operation; the capacity of the plants being seriously affected, the waste liquor being high in ammonia content and the steam-ammonia stream low in ammonia concentration, causing trouble in the saturator.

Prior to the admission of steam to the still for heating purposes it is important that all the trays should be sealed as well as the steam pipe or jets in the liming chamber and the fixed still. The heaters and coolers should also be filled with gas liquor and water respectively so as to prevent possible fracture of the pipes when the cold liquor is first introduced. Provision should be made for venting the air from the still. A suitable arrangement is to take a 1½in. wrought iron pipe from the cast iron pipe leading from the still to the saturator, and provide this with a cock, allowing the discharged foul air to be emitted to the atmosphere.

The practice of venting a still inside the still-house is a dangerous one and should be discountenanced.

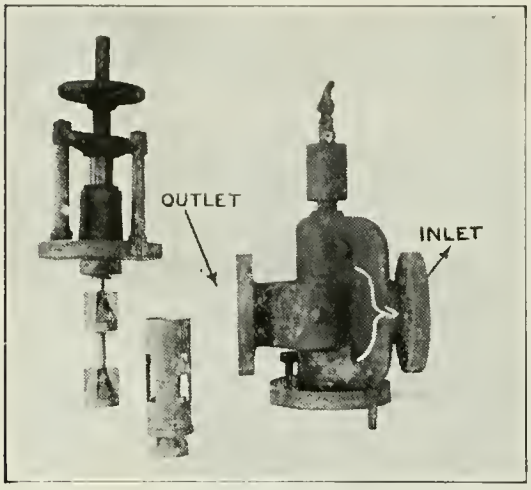


FIG. 44.
Reducing Valve and parts.

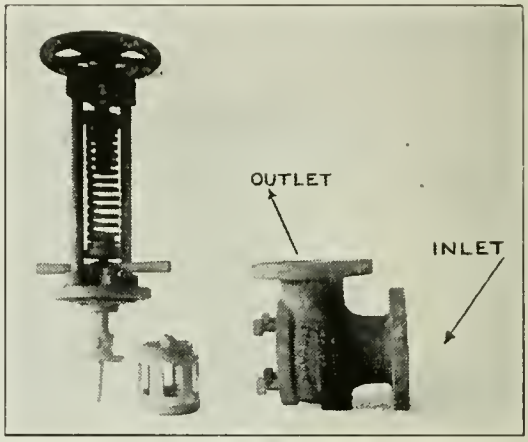


FIG. 46.
Photograph of Gas Liquor By-Pass Valve.

The gradual admission of steam to the still is important, particularly with the types of distillation apparatus which consist of a series of double-flanged pieces which are heavy in character. Such stills must be cautiously heated, otherwise the irregularities of expansion will cause fracture.

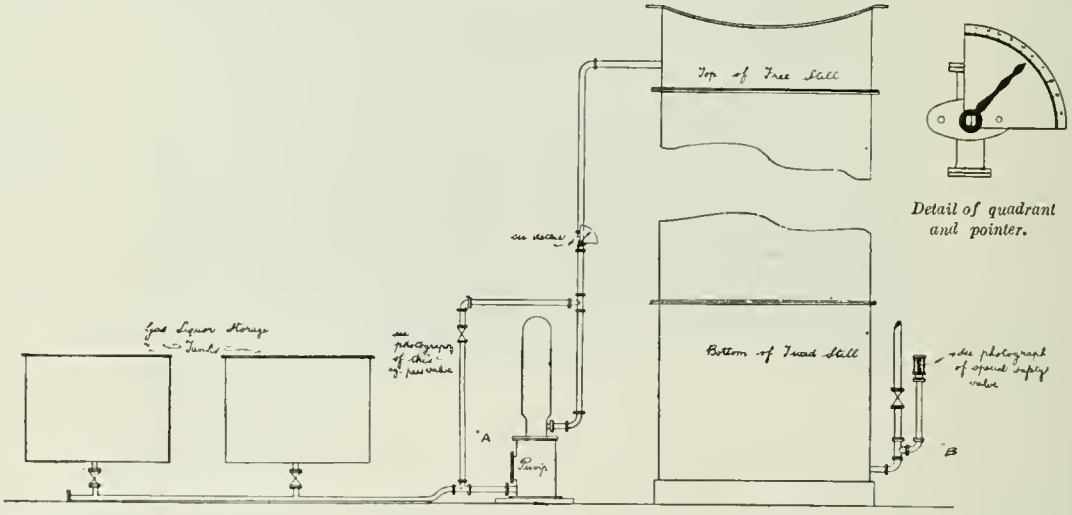


FIG. 45.
Diagrammatic Sketch, showing arrangement for (a) maintaining constant feed of gas liquor, and (b) safety-valve arrangement.

With a new still of tolerably large capacity at least twelve hours should be allowed for raising the temperature to the requisite degree for the admission of the gas liquor.

Thermometers should be provided, preferably fixed in suitable oil pockets, on the liquor inlet

to the preheaters and on the outlet to the still, and similarly thermometers should be fixed on the waste gas inlet to the coolers and on the outlet.

It is also desirable to arrange a centrally situated gauge board as shown in Fig. 47, so that the boiler steam pressure and the reduced steam pressure, as well as the pressure of the liquor being pumped to the stills and the resistance thrown by the passage of the waste gases through the heaters and coolers and subsequent pipe lines, can be observed without difficulty by the operator of the plant.

It is equally important that each of the stills in the case of category A type of plant, and the columns in the case of categories B and C should be provided with an efficient safety valve. Fig. 45 shows a suitable point at which the valve can be fixed, and Fig. 48 shows the constituent parts of the suggested safety valve, which is of a simple spring type and can be easily adjusted and set to relieve at any predetermined pressure.

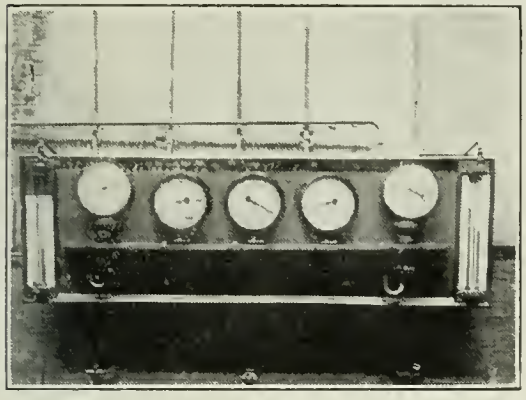


FIG. 47.
Photograph of Gauge Board.

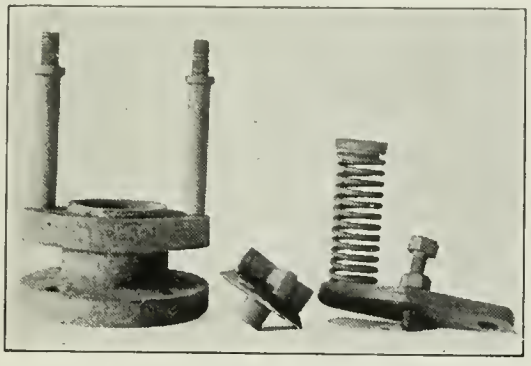


FIG. 48.
Photograph of Safety Valve.

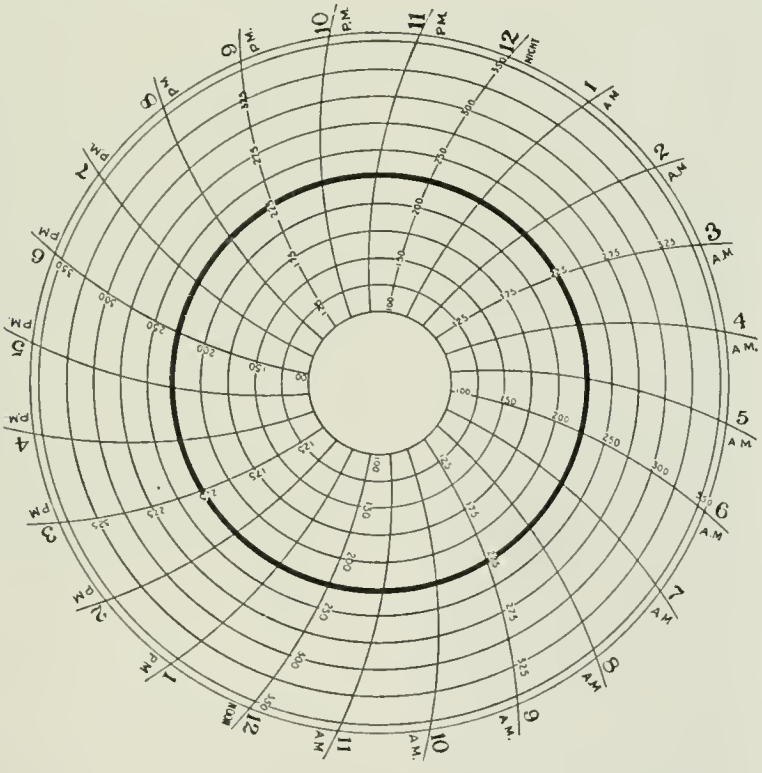


FIG. 49.
Continuous thermagraphic record of the temperature of the effluent liquor leaving a gas liquor still.

The heating of a still after temporary stoppage need not occupy more than four hours. After the top portion of the still has become warm, the cock of the vent pipe can be closed and the steam sent forward through the saturator, the cracker pipes in which have been sealed with mother liquor. It is important that the devil liquor

outlet cocks should be opened immediately the heaters become warm in order to allow of the automatic withdrawal of condensation which must arise, due to the cooling influence of the gas liquor and water with which the heaters and coolers are filled.

Prior to feeding the still with gas liquor it is wise to pump, or gravitate, milk or cream of lime to the still, so that by the time the gas liquor is introduced, there is a complement of boiling milk or cream of lime in the liming chamber.

The raising of the temperature of the apparatus to this point is usually done with steam at boiler pressure, but immediately the liquor feed is commenced the reducing valve should be brought into operation by removing one or more of the weights on the spindle. The next step is to direct attention to the passage of the limed liquor to the fixed still, the connecting pipe to which is usually provided with a cock so that samples can be drawn from time to time. As soon as the limed liquor enters the fixed still a further and final adjustment of the steam reducing valve and the steam inlet valves to the stills is necessary.

Temperatures at the essential parts of the plant should be taken at regular intervals and should be recorded. Equally the liquor feed gauge board should be watched very carefully so as to make certain that a uniform feed is being obtained. Records of the liquor fed to the still should be made half-hourly.

It is usual to connect a cast iron pot to the fixed still, and to provide such pot with two vertical pipes, one of which is carried almost to the foot of the pot and which is provided above the pot with a glass tube suitably connected to give an indication of the working level of the still, the other pipe being attached to the cover of the pot in order to serve as a safety valve.

One other point of importance as affecting the operation of gas liquor stills has reference to the necessity for a uniform feed of devil liquor along with the gas liquor. It has been found that an indication of the uniformity of conditions is furnished by taking a continuous temperature record of the waste liquor leaving the still. A typical chart taken under such conditions is shown in Fig. 49.

A continuous sample of the waste liquor leaving the still should be taken and a definite standard adhered to in this connexion. Experience suggests that 0.015 per cent. NH_3 in the waste liquor is an economic strength, as working below this limit simply resolves itself into the recovery of an exiguous amount of ammonia at the expense of labour in the operation of the plant. The devil liquor should be tested periodically from the points of view of its freedom or otherwise from ammonia and its volume. The presence of ammonia indicates that leakages are occurring, and these should be arrested at the very outset. An unduly large volume of devil liquor suggests a high consumption of steam in the distillation of the liquor, or otherwise the leakage of water in the coolers.

Early indications are furnished of the fouling of the stills by the necessity for reducing the volume of steam being introduced thereto, and it will be found, other things being equal, that the effluent liquor gradually increases in point of ammonia content. If tar accidentally becomes mixed with the liquor, in order to prevent this adversely affecting the working of the still, the preheaters and pipe lines to the still should be discharged, such discharged liquor being returned to a tank for separation or other suitable treatment.

In concluding these observations the author desires to summarise his views as to the direction in which progress in the design of ammoniacal liquor stills is likely to proceed in the future.

Designers must concentrate their attention and energies to the evolution of the elements of a still which will be economical as regards steam consumption. Data must be arrived at as to the relative value of attenuated bubbling, as compared with time contact of the liquid and the vapours as a factor in aiding equilibrium. Obviously distillation cannot proceed until the vapour pressure of the ammonia in solution in the trays is in excess of the vapour pressure of the ammonia in the vapours.

The author is led to the belief that such a form of bubbling device can be designed as will not only assist in promoting optimum bubbling, with maximum impact between the respective series of bubbles so formed, but that such an arrangement need not necessarily appreciably reduce the factor of time contact of the liquid and vapour. Such time contact can obviously be increased by allowing a tolerably large vapour space above the liquid in the trays.

Optimum bubbling is a function of a definite vapour velocity, which, when exceeded, will hold up the liquid and prevent contact between the vapour and liquid—in other words, will defeat its own object. This definite vapour velocity will differ in each compartment of the still, by reason of the varying vapour volumes, and instead of as at present the serrations, or perforations, of the bubblers being uniform in size, these must be modified in accordance with the varying volumes indicated.

As regards the utilisation of the surplus heat leaving the saturator, it is evident that the present arrangement of plant as generally known must be improved in order that more satisfactory conservation of the heat available can be ensured. There are several possibilities in this direction which must occur to one on careful consideration of the conditions obtaining.

Finally the author wishes to express his thanks to those chemical plant manufacturers and gas engineers who have kindly placed drawings and photographs of their stills and accessory plant at his disposal, without which the paper must obviously have been less comprehensive in its scope and data.

He also wishes to express his thanks to Dr. Carpenter and Mr. E. V. Evans, F.I.C. (the Chief of the Chemical Department), for their permission to publish certain experimental and other data embodied in the paper, and for their helpful guidance at all times.

His thanks are likewise due to Messrs. O. W. Weight, F. C. Snelling, and C. E. Parr for their assistance in the preparation of several of the drawings and diagrams, and for their helpful suggestions.

Discussion.

The Chairman said he was sure they had all listened very attentively to Mr. Parrish, and he hoped also that they had read the advance copies of the paper prior to the meeting. He would not speak on it himself at the moment, but would say something on the paper at the end of the discussion.

Mr. Charles Cooper said that in the paper it showed that the author had a radiation loss on the sulphate still of about 3000 B.Th.U., and on the heat exchangers of 70,000 B.Th.U., with presumably approximately the same temperature differences and proportionate areas. This was a matter upon which he would like to have a word before the discussion ended. In the author's remarks on various designs of columns a common feature was mentioned, *i.e.*, the large number and area represented by the serra-

tions compared with the diameter of the gasway. The connection was one which could better be appreciated mentally than stated in precise values. The penalty of not true levelling when erecting a column was, no doubt, more heavily felt in such cases. The author had hardly touched upon a matter that seemed to him (Mr. Cooper) to be quite important, that was, the entrainment of liquid from tray to tray upwards, and the manner in which it affected the efficiency of removal for a given steam consumption. Where entrainment took place in sensible quantity, two effects were felt. One of these could best be appreciated by stating the limiting case, which was what would obtain if it were possible to blow the whole of the liquor out of the still; a single-stage action would be produced, and any given proportion of removal would be very wasteful in steam compared with a multiple-stage treatment of the same gross capacity. In a more general form, this proposition could be stated in this way: that in any countercurrent process carried on in interrupted stages there was a downward limit of the number of stages which must be provided to produce a definite proportionate stripping along with any desired richness of product (in this case a steam-ammonia mixture). The first effect of entrainment was to blur the sharpness of the "cut" between stages, and the practical result was the same as if the gross capacity provided had been divided into a less number of sections. The second effect was one of dilution of the liquid in any section by the addition of weaker material from below, therefore producing the result that a greater quantity of liquor of lower strength was actually being stripped. These two considerations dictated at some point a limit on the amount of turbulence desirable in such a column. That considerable entrainment could take place was seen in the provision of a void, and frequently a trap for drops, at the outlet of the column. The lime-charged liquor was also liable to froth, but, of course, there could be no sharp demarcation between frothing and entrainment. This led him to suggest that the prevalence of large bubbling areas in various designs had possibly arisen from the manufacturer finding that the advantage of splitting his available area or volume into a large number of sections was nullified by the conditions stated, and he had compromised on empirical results, but at different values. With respect to designing for minimum steam consumption, the limiting conditions appeared to be these: that, once having all liquor admitted at the boiling point for the still pressure, the only heat absorptions were those represented by the dissociation of ammonia salts, and, of course, the hydrate, the radiation losses, and the elevation of boiling point due to rising pressure as the liquor travelled downward. All these were represented by condensation and loss of superheat, if any, of the steam, and presumably were irreducible or subject to proper treatment. They had further to provide a vehicle to effect the release of the ammonia, and the steam employed here had a minimum value represented by such values as Piron's, quoted by Mr. Parrish. The partial pressure of ammonia in solution of its compounds at about 100° C. was not the only quantity which appeared to matter, according to certain figures which he had examined. Without attempting to go too deeply into this aspect of the matter, the conditions in the column to which those figures referred showed that the actual consumption was about 1½ times the minimum quantity required merely as a vehicle for the ammonia; and a part of the excess was represented by the losses to be made up in the column. The consumption was about the same as that shown on the thermal balance sheet, but the distribution was different, a greater proportion being admitted to the fixed still. F. H. Wagner, in his book on "Coal-Gas Residuals," had stated the heat absorption per lb. of NH_3 dissociated in the still from salts as 1880 B.Th.U. Hence, for 15·5 lb. NH_3 an absorption of 29,000 B.Th.U. was to be allowed, equal to 30 lb. of steam, which was 13 % of the total steam consumption.

Dr. E. W. Smith said that as the paper was an attempt to show that increased efficiency should be obtained in sulphate production, he would have liked some very strong reference to what gas works might do for themselves before the ammonia liquor was sent to the distillery, in assisting to reduce the cost of sulphate production. It was an old suggestion, but now that the new gas Act had come into force, and the therm basis was standard, and the illuminating power of the gas was no longer necessary, and benzol was, or should be, extracted from all coal gas, it was obvious that any gas undertaking that did not employ an acid catch for its ammonia extraction prior to the purifiers was wasting money and energy. If they considered that most gas works supplied liquor of average strength of perhaps 8 ounces, it would be quite an easy thing to produce a liquor of 15 to 16 ounces with an acid catch, and in that way eliminate at least one-third of the ammonia scrubbers. It would be a good thing to the distiller of liquor if that large amount of water could be eliminated, and now that it was possible and really workable he felt that it would be well worth concentrating on what might be done before they came to the stills, and before it was necessary to do any standardising.

Mr. W. R. Sibbald said that, as a matter of fact, in supplying steam to a still of this type it was found that by reducing the size of pipe between the superheater and the still a very considerable economy of steam, amounting to almost 25 per cent., was effected. If the question of the size of pipe had been taken into account, economy would have taken place much sooner. Another point was that it seemed to be common practice to use serrations in the hoods. He supposed the idea was that automatically the bubbles passing through would have their minimum size. As a matter of fact, in using a serrated hood, he had found that such a hood was very sensitive. If there were a slight slope, or if the hood were slightly displaced, or if there were some fault in the design, it caused it to tilt slightly to one side, and the steam was all discharged on a narrow portion of the periphery of the hood and gave very large bubbles. The result of that was that there was a great deal of entrainment, whereas if the bubbles were reduced to a minimum equilibrium was established in the minimum time. In considering this difficulty he had tried different types of hood, and as an alternative to the ordinary one he had several rows of holes, 1/16th-in. in diameter, drilled all round. That gave a fine division of the gas and brought about equilibrium much more rapidly. The fault of that, however, was that such a design was very easily choked up, so a second alternative was adopted, in which, instead of 1/16th-in. holes, a slot of about 1-in. depth and 3/32nd-in. width was tried. A series of such slots was cut with a hack saw, and so far as he could gather—it was rather a difficult matter to test—the sub-division of the gas was almost exactly the same as with the holes. It seemed to him that this was not a very minor matter, because if the gas equilibrium were established rapidly it meant that the number of chambers in the still could be reduced, and consequently the total surface area of the still would be reduced, and the radiation loss factor, which was put at the high figure of 21 per cent., would be reduced to a minimum.

Dr. L. F. Goodwin said there was an obvious misprint in the radiation loss in the heat entering the still at the bottom of page 8, which was given as 0.3 lb., and ought to be 3lb. Nevertheless, even with that correction, a radiation loss of 0.7 per cent. appeared to be very small. The only other point he

wished to bring up was the question of intimate contact between the bubbles of the liquid, which was the same type of problem in the column still or saturator as it was in any other type of absorption. With a serrated hood the slightest change in level between any of the slots with regard to the rest of the liquor led to unequal bubbling. If there was a certain minimum depth to overcome a certain minimum amount of head, it produced more uniform bubbling all round. One final point was that he should think small traces of oil, or an oil film, in any liquor of that sort would have a great deal to do with intimate contact between the gas and the still.

Mr. A. G. Byard (France) said that he was concerned with the installation of a Wilton ammonia plant, and it was attached to two steam boilers. These steam boilers were certainly not overworked at the time the plant was installed, but they were overworked when the plant was put into operation, and the plant worked satisfactorily while the rest of the works stood still. The poor gas manager asked what he was to do, and he told him he must either put in another boiler or else stop the works altogether. About this time a friend of his asked him (Mr. Byard) if he could introduce into Spain some superheating apparatus. He arranged that the experimental apparatus should be installed at his works. They started with low pressure in the boilers, at about 90 lb., or 6 atmospheres, and the pressure of the steam, when introduced into the ammonia still, was about 14 lb., or, say, 1 atmosphere. He did not think that anyone had thought of superheating the steam before then, but it was done, and the gas works went on their way quite satisfactorily, with the result that one boiler was found necessary instead of two, and instead of stopping the gas works, as before. Therefore, he thought that the question of superheating steam was well worthy of consideration.

The Chairman said he would call upon Mr. Parrish to reply. But before doing so, he wished to make a few remarks himself. In the first place, he wished to compliment Mr. Parrish on the paper for one special reason, and that was that it was the first paper, he believed, that had been published in this country—he did not know about the Continent—in which some attempt had been made to apply scientific principles to the design of, or to ideas of designing, a still for separating ammonia from gas liquor. They called an apparatus or a vessel for treating gas liquor a still, but personally he did not think it was quite the right word. They were dealing with rather a peculiar condition of affairs. They had a liquid in which the gas was dissolved, and what they were doing was to devise an apparatus to boil out that gas. If they could do that without distilling the water they would have an ideal apparatus. That was what they were aiming at, and the more they had that in view—and he was sure Mr. Parrish had it in view, because he was talking so much about the supply of steam—the more perfect piece of apparatus they would have. With regard to the serrated hoods, he would like to give a little piece of practical experience of his own. He had distilled gases in many types of apparatus, and finally was induced to put in a Continental still, or, as it was called, a Continental dissociator. That was a Mallet apparatus, and with that still the whole of the steam required to make a ton of sulphate of ammonia was obtained with $9\frac{1}{2}$ cwt. of coal. That was an actual figure, from a week or a fortnight's test, and included the whole of the steam used on the plant, except the steam for pumping the liquor into the store bin. Therefore, he thought the question of serrations was a little too accentuated, but possibly some of them might have had experience of a still of that type and could give their results. Mr. Parrish had given some very interesting diagrams, which he (the Chairman) was sure would be of very great service to anyone engaged in designing stills or working them.

Mr. Parrish, in reply to the discussion, said it was obvious that Mr. Cooper understood the fundamental principles involved in the distillation of ammoniacal liquor, but he was afraid that he had misunderstood the paper in connection with one or two points that he had raised. So far as the still was concerned, he had attempted in the paper to itemise the loss involved, and in that connection, despite what Dr. Goodwin had said, the loss from radiation was only 0·7%. He did not, however, wish them to confuse the loss via radiation as between the still and the preheaters. The preheaters were placed outside purposely with a view to assisting in the cooling of the steam and waste gases which came from the saturator, because there was such a volume, quite apart from raising the ammoniacal liquor to boiling point, that water had to be used in order to dissipate that heat before the waste gases could be sent forward to the spent oxide burners for combustion. Mr. Cooper had directed his remarks to the question of entrainment. In connection with all the experiments that he himself had conducted as regards the design of the elements constituting an ammoniacal liquor still, he had had the question of optimum velocity well in mind. There were, however, certain figures which could not be disclosed in connection with these experiments, because they were fundamental to any new design which might possibly be patented. But he had not overlooked the question of entrainment, and he could assure Mr. Cooper that, so far as the stills in the paper were concerned, there were very few which were calculated to afford much difficulty from the point of view of entrainment, having regard to the definite vapour velocity which they gave. In point of fact, the steam could be increased by over 300%, and even then there would not be any difficulty from entrainment, so that that aspect had not been overlooked. The question of turbulency was a very important matter, as he had attempted to demonstrate. Of course, it must be considered in relation to that maximum velocity which did not create entrainment. There was also another question which affected turbulency, and also concerned entrainment, and that was the question of constant steam. If Mr. Cooper had had any experience of stills throughout the country, he would have found that there were very few people who understood the reducing valve, and singularly few who did understand the reducing valve who even attempted to control the working of it so that they could get anything approaching constant steam conditions. If they were rushing steam into a still in gulps they would get turbulency of motion, quite apart from the question of the design of the particular element involved. Dr. Smith's remarks were very pertinent, and he himself was still investigating the question as to the maximum strength at which gas works ought to operate their scrubbing plant, in order that it might be reasonably economical, both from the point of view of the gas works, and from the point of view of the chemical works which had to deal with ammoniacal liquor. Indeed, if they looked at the curve (Fig 15) it would be seen that it was only necessary to continue that curve with higher concentrations, up to, say, 5% of ammonia, in the crude liquor, in order to see precisely what the economy in steam was likely to be. There was one difficulty so far as the acid catch was concerned when it was interposed between the scrubber and the purifiers, and it was a difficulty which quite a number of gas works which had worked direct plants for the absorption of ammonia had been face to face with, and that was the question of the entrainment of the vesicular acid, which caused souring in the purifier through reaction of the acid mist with the oxide

of iron, forming sulphate of iron. The oxide of iron in the purifiers must be alkaline, and it was impossible to purify the gas until such soluble iron, which was caused by the reaction of vesicular acid and oxide of iron in the purifier, was neutralised, and the ferrous sulphate was decomposed. He did not say they could not use an acid catch and obviate entrainment, but there was a difficulty which needed to be carefully considered in connection with any such proposal. With regard to Mr. Sibbald's remarks, he had not suggested that steam consumption was a minor matter when introducing the paper. What he had said was that steam consumption, so far as these elements were concerned, was a minor matter, and that the major question was the utilisation of the heat leaving the saturator. All the remarks that Mr. Cooper had made concerning 15% being the maximum possible saving of steam consumption were directed to the type of sulphate of ammonia plant as existing to-day, and they were not relevant to the plant which he himself had attempted to visualise in his introductory remarks as the type of plant which would be evolved as the result of investigations of this character. Mr. Sibbald had alluded to the form of the serrations, but if he referred to the photograph (Fig. 6) he would find that the bubbler there was provided with about 1000 holes. If he (Mr. Sibbald) put a magnifying glass to that photograph he would find that the central holes were about twice the size of the outer ones. That bubbler had been designed, not haphazard, but as the result of a good deal of experience in connection with this question of bubbling, which was closely bound up with promoting equilibrium with minimum steam consumption. Dr. Goodwin had referred to the depth of bubble and the question of alignment. So far as he personally was concerned, extraordinary care was taken in aligning sulphate of ammonia stills. He was able to get them dead level, and he believed in the foundations being such that there could not be any possibility of subsidence. He could definitely ensure that there was absolute uniformity so far as the slot of the hood or bubbling device was concerned. It was quite wrong to increase the depth of the slot unnecessarily, because it would be found that it added to the steam consumption. The smaller the depth of the seal, consistent with ensuring uniformity, the better from the point of view of steam consumption. As regards oil, there was no ammoniacal liquor in the country that was absolutely free from oil. Those people who were settling best were invariably the works which introduced a certain amount of hydrocarbons specifically lighter than the ammoniacal liquor itself, and these hydrocarbons were usually oils of the creosote fraction, so that in connection with distillation one was always faced with this question of oil, and, indeed, received some little assistance from it. He was interested in the remarks of Mr. Byard, and had himself not only looked into the question of stills as produced in this country, but had given consideration to the patent specifications of the stills produced throughout the world, and he could assure them that chemical engineers had yet a long way to go before they attained perfection in the matter of design. It had been said that there was no finality to thought or achievement, and he was convinced that so far as ammoniacal liquor stills and their design was concerned, finality had by no means yet been reached.

On the motion of the Chairman, a vote of thanks to Mr. Parrish was carried with acclamation.

TAR DISTILLATION.

By W. A. WALMSLEY.

Coal-gas tar is a highly complex mixture of organic compounds. Its specific gravity and composition vary according to (1) the method of carbonisation of the coal, (2) the class of coal used, and (3) the temperature of carbonisation of the coal.

Up to about ten years ago town's gas was made almost exclusively in horizontal retorts at high temperature, and the resulting tar was thick and heavy. Horizontal-retort tar has been fairly thoroughly investigated by a number of workers, and its constituents proved to be of an aromatic character. On distillation, it yields approximately 60-70 per cent. of pitch of good merchantable quality, and 30-40 per cent. of oils containing aromatic hydrocarbons of the benzene, naphthalene, and anthracene series, phenols and phenol homologues, pyridines, etc.

With the introduction of the vertical retort, a radical change occurred in the tar. The specific gravity was lower, and the tar of a more oily nature. Glasgow vertical tar yields on distillation 40-44 per cent. of pitch and 56-60 per cent. of oils.

Let us consider for a moment the method of formation of these two tars. The tar vapours in horizontal retorts have to pass through the hot outer crust of the charge, and also to some extent along the hot walls of the retort. Breakdown occurs, and the resulting tar is benzenoid in character.

In vertical retorts the earlier portions of tar vapours are provided with a cool means of escape from the retort through zones at a lower temperature than that at which they are formed. It is even possible that they have some solvent action on the coal in these lower temperature zones, and pick up, in passing, some of the primary constituents of the coal stuff itself. The tar resulting from such conditions is paraffinoid in nature, and is known as low-temperature tar. The later portions of tar vapours, however, are subjected to some extent to the conditions that appertain in horizontal retorts and give benzenoid tar.

Vertical-retort tar may therefore be looked upon as a mixture of high- and low-temperature tars. No thorough investigation has been made of vertical-retort tar. It contains less benzol than horizontal tar, practically no naphthalene, a large percentage of compounds extractable with caustic soda, containing very little real phenol. These compounds would appear to approximate to the phenolic substances extractable from wood tar creosote, which contains guaiacol and coerulignol.

Some recent experiments carried out by us appear to indicate the presence of even trihydric phenols in the higher boiling portions. They show great affinity for oxygen, absorbing it very readily in the same way as pyrogallol.

The following figures serve to show the difference in distillation range between the tar acids obtained from vertical and horizontal retort tars. The whole of the oils up to pitch were extracted with caustic soda, and the resulting tar acids dried and then distilled.

Horizontal-retort tar.

The crude dry tar acids obtained were equivalent to 9% by weight of crude tar, and had the following distillation range, using a Wurtz flask:—

Drop, ...	186° C.
45·2% at	200° C.
69% ,, ...	210° C.
72·5% ,, ...	220° C.
81% ,, ...	230° C.
84·5% ,, ...	240° C.
88% ,, ...	250° C.

Vertical-retort tar.

The crude dry tar acids obtained were equivalent to 13·2% by weight of crude tar, and had the following distillation range, using a Wurtz flask:—

Drop.	185° C.
10% at	200° C.
28% ,, ...	210° C.
45% ,, ...	220° C.
56% ,, ...	230° C.
63% ,, ...	240° C.
78% ,, ...	280° C.
84% ,, ...	300° C.
92% ,, ...	310° C.

Distilled above 260° C. ruby red in colour. Gave highly viscous fluid when cooled.

It was thought by us that tar acids obtained from vertical-retort tar might approximate to some considerable extent to the high coefficient acids obtainable from blast-furnace creosote, and the comparison between the distillation ranges and physical properties was made, with the following results:—

Distillation tests of tar acids.

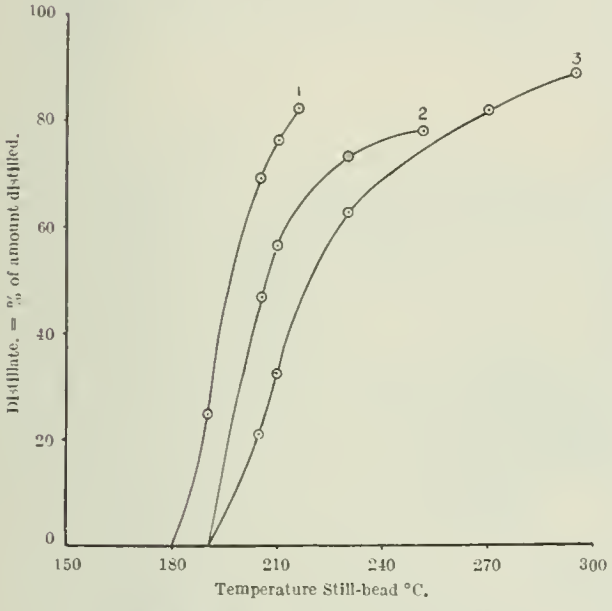
Fraction.	Dalm. cr. naphtha.		Dalm. mid. oil.		Blast-furnace tar acids.	
	% between these temperatures.	% up to last-named temp.				
	a.	*b.*	*a.*	*b.*	*a.*	*b.*
–190°	24·6	24·6	—	—	—	—
190°–205°	24·8	69·4	47	47	21·4	21·4
205°–210°	7	76·4	9·7	56·7	11	32·4
210°–216°	5·7	82·1	—	—	—	—
210°–230°	—	—	17	73·7	30·7	63·1
230°–252°	—	—	4·5	78·2	—	—
230°–270°	—	—	—	—	19·4	82·5
270°–295°	—	—	—	—	7·3	89·8

Tar acids previously dehydrated where necessary.

Fraction.	A. Sp. gr. 15·5° C.	A. Refractive Index. Mean D line 25° C.	B. Sp. gr.	B. Refractive Index.	C. Sp. gr.	C. Refractive Index.
–190°	1·055L	1·5435	—	—	—	—
190°–205°	1·036L	1·5385	1·038L	1·5362	1·031L	1·5385
205°–210°	1·022L	1·5375	1·020L	1·5370	1·021L	1·5330
210°–216°	1·020L	1·5370	—	—	—	—
210°–230°	—	—	1·013L	1·5350	1·011L	1·5325
230°–252°	—	—	1·047N	1·5500	—	—
230°–270°	—	—	—	—	1·038N	1·5330
270°–295°	—	—	—	—	1·066N	1·5730

Note.—L after the specific gravity indicates that the fraction gives Liebermann reaction for phenols.
N indicates that it does not.

Fractional Distillation of Tar Acids.
Young's Evaporator Column. Twelve Sections.



1. Tar Acids from Crude Naphtha. (Vert.-retort Tar).
2. Tar Acids from Middle Oil. (Vert.-retort Tar).
3. Blast Furnace Tar Acids.

The following, taken from monthly distillation and yield statements from two separate works, one distilling horizontal-retort tar, and the other distilling vertical-retort tar, illustrates the difference in specific gravity, percentage of volatile products, and percentage of pitch obtained, during the primary or crude distillation of the tar.

	Vertical tar. Specific gravity 1·091.		*Horizontal tar.* Specific gravity 1·175.	
	Sp. gr.	% Yield.	Sp. gr.	% Yield.
Ammonia water	1·025	4·0	1·003	3
Crude naphtha	·946	2·59	·925	1·34
Light oil	—	—	1·007	6·02
Middle oil..	·996	30·91	1·025	6·18
Creosote	1·053	20·09	1·045	16·32
Pitch	—	41·47	—	66·56
	Total	99·06	Total	99·42

	Vertical tar.	*Horizontal tar.*
Viscosity (Redwood).	345 secs. at 100°F.	610 secs. at 100°F.
Free carbon	1·19%	11·3%
Calorific value	16,120 B.Th.U.	16,150 B.Th.U.
Pitch volatile matter ..	69·9%	53%
Fixed carbon	30%	47%
Free carbon	5%	25%
Ash	0·1%	0·1%

It is only proposed here to give a brief outline of the methods adopted in tar distilling, and to describe in more detail the type of plant adopted, and its layout at the Provan Works of the Glasgow Corporation.

Tar distilling may be roughly divided into two parts:—(1) The primary or crude distillation, and (2) the refining operations. The crude distillation is carried out in closed stills, and consists in evaporating from the tar a certain amount of its volatile matter, condensing this volatile matter, and separating it into fractions, leaving behind in the still a residue of pitch of good merchantable quality. The number of fractions made from the volatile matter varies, but the following may be taken as fairly average practice:—

(1) Crude naphtha, containing benzene, toluene, xylene, carbolic acid, etc.

(2) Light oil, containing mainly carbolic and cresylic acids with some naphthas, and in the case of horizontal tars, naphthalene.

(3) Carbolic oil, containing tar acids, mainly cresols.

(4) Light creosote oil.

(5) Heavy creosote oil or anthracene oil, containing anthracene, etc.

REFINING OPERATIONS.

All subsequent operations are classed under this head.

(1) *Carbolic extraction.* Fractions 1, 2, and 3 are washed with a 10 per cent. solution of caustic soda, prepared from lime and sodium carbonate. The caustic soda removes tar acids as their sodium salts. Treatment of the sodium salts with carbon dioxide liberates the tar acids, and regenerates sodium carbonate, which is recausticised with lime, and used over again. The crude tar acids are purified by fractional distillation.

(2) *Treatment of washed oils.* The washed oils from fractions 1 and 2 are distilled. The distillate known as "once-run naphtha" passes to the benzol refinery for further treatment. The residue, along with fraction 3, is cooled in open pans and deposits crystals of crude naphthalene. The clear oil is drained from the naphthalene (which in our own case is sold as such), and goes along with fractions 5 and 6 to creosote storage.

(3) *Treatment of "once-run naphtha."* The "once-run naphtha" is first washed (in mechanical agitators) with a 25 per cent. solution of sulphuric acid, and allowed to settle. The mixture separates into two layers; the lower layer containing pyridine bases, as their sulphates is run off. The pyridine is recovered by treatment with ammonia with formation of ammonium sulphate and liberation of the pyridine. The pyridine-free naphthas are then washed as follows, allowed

to settle, and the lower layer run off in each case:—(1) With about 5 per cent. R.O.V. 168° Tw. to remove thiophen, etc., (2) with water (without agitation); with water (with agitation), to remove as much acid as possible. (3) With caustic soda or sodium carbonate to neutralise any acid remaining. (4) With water to remove any excess caustic soda. The washed naphthas are then subjected to fractional distillation by means of steam in stills with efficient fractionating columns, and analysers, to separate benzene, toluene, xylene, etc.

Note.—The above only involves the treatment of tar as carried out in plant already erected or in course of erection at the works.

In Glasgow, the Corporation have four Chemical Works for the treatment of tar and ammonia. Three of these were taken over from private contractors two years ago. It was necessary to lay down some working scheme, and the author decided to continue crude distillation of the tar at three of the works, and concentrate on one, viz., Provan, for all refining purposes.

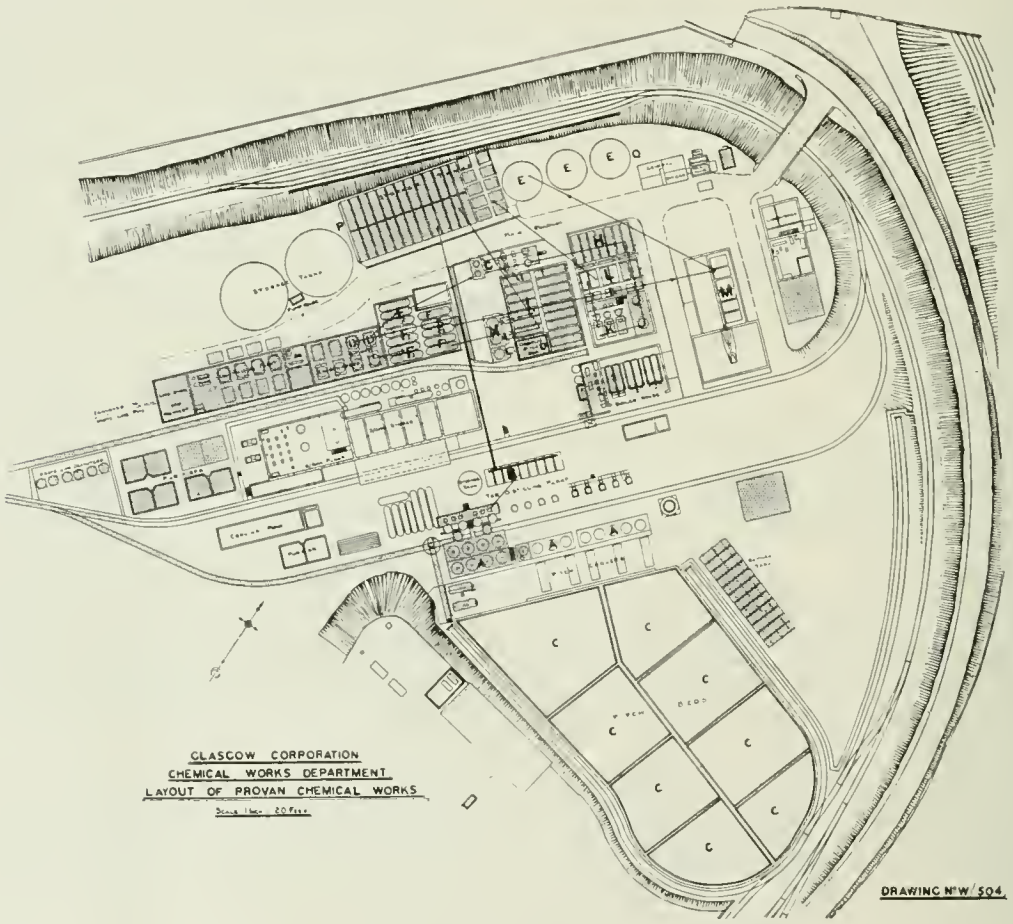
It is obviously more economical in regard to manufacturing costs, and also in regard to capital outlay, to have one really good refining plant, than to put down four. Another most important point is that there is only one staff of chemists looking after refining operations, and it is, therefore, easier to turn out a standard product.

The importance of layout in the efficient running of works is too well known to need emphasis here.

Railway sidings. Starting at the North, the first, or main, line skirts the works on North, East, and South, and the first line from it is a switch line for the loading of all oils leaving the works by rail.

The next line branches off on the East, divides and turns West on line passing in front of the boiler house and sulphate of ammonia plant, and the other in front of the tar stills. These finally join together and turn North to meet the main line at a point further West not shown on the plan.

Between these lines and the main road is the siding with blank end for the causticising and



Layout of Works.

Block Plan of Provan Works.

carbolic plant, etc. On the South, the pitch beds are enclosed on three sides by railway lines, which also serve for the loading of road tars in railway tank wagons. A further line to the S. and E. with blank end, serves for the loading of tar sent out in barrels. All oils sent out by road are loaded at a raised bank alongside and parallel with the main road.

Flow sheet of materials through works. The tar is distilled in the stills, A. The distillates are collected in the receivers, B. The pitch passes to the pitch coolers, and thence to the pitch bay, C. The oils from B for carbolic extraction pass to washers, D, the remainder to oil storage, E. From D. washed crude naphtha and light oils run to receivers, F., and pass from there through naphtha stripping plant, G. The distillates from G—"once-run naphtha"—run to receivers, H, at the other. This applies also to the layout of the sulphate of ammonia plant.

Fig. 2 illustrates the type of still in operation at three of our works.

(A) Tar still, which is simply an egg-ended boiler 9ft. dia., 17ft. 6in. on the straight, and 23ft. 6in. overall.

(B) Gusset stay brackets—6 in number—riveted to still. These support the still by resting on the brickwork setting.

(C) 4in. charging main.

(D) Deadweight safety valve.

(E) Dip cock.

(F) Manhole (in this connexion, the correct type is the internal type used on mudholes of steam boilers).

(G) Vapour outlet.

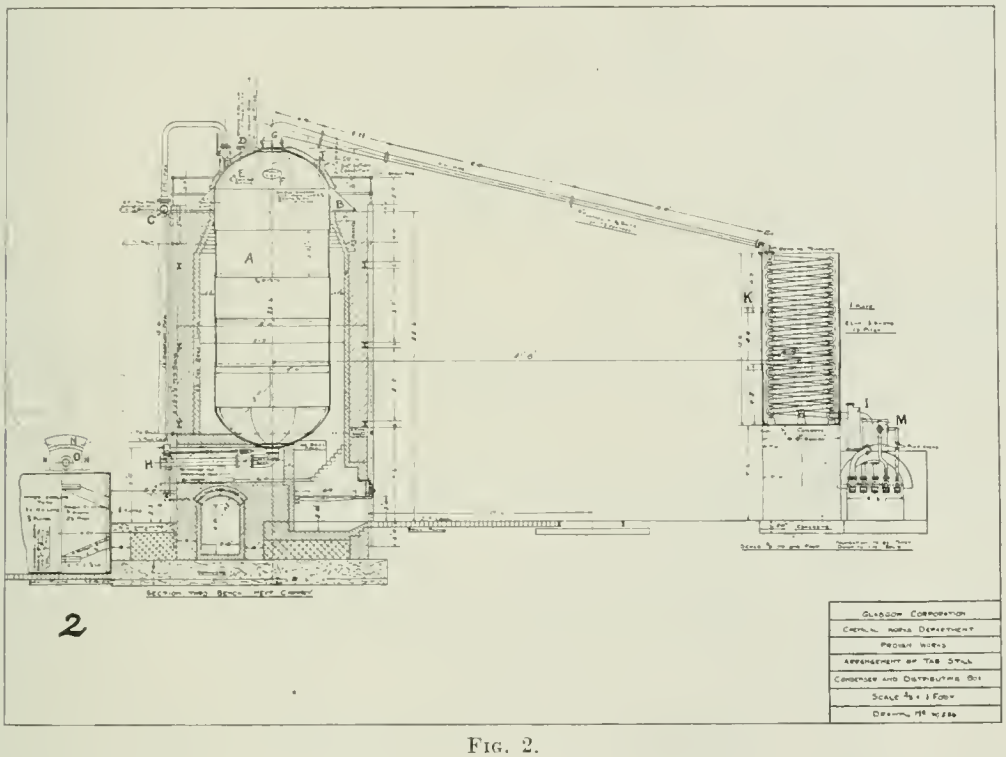


FIG. 2.

residue to receivers, I, and thence to naphthalene cooling pans, M.

Once-run naphtha from H is pumped to washers, J, and thence into benzol refining stills, K. The distillates pass to receivers, L, and residue to naphthalene pans, M. From D washed carbolic oil runs to receivers, F_2, and from these passes to M.

Crude carbolic and cresylic acids from the carbonators run to receivers, F_3, and pass from there to carbolic stills, N. The distillates from N go to receivers, I_2, the residue to carbolic pitch bed O. Finally all products from I_2, L, and M are pumped to oil storage, E, from which all classes of oil are loaded for road or rail. E comprises all oil storage tanks between P and Q.

On the refining side, the object has been to get a straight flow line through the works, with finished products at one end, and waste materials

(H) Pitch run-off pipe with steam-jacketed gland cock.

(J) Steam connexion for steam distillation.

(K) Condenser tank and coil.

(L) Steel box.

(M) Dividing box for separating fractions.

The method of setting is easily followed from the drawing. It will be noticed that the pitch outlet pipe is protected from the heat by the fire brickwork, N, and the arched passage, O.

All crude tar contains a certain percentage of entangled ammonia water. The removal of this ammonia water, which comes over along with the crude naphtha fraction, is the most troublesome part of the distillation. The still must be fired very cautiously, until all ammonia water has been driven off, otherwise frothing takes place, and a considerable quantity of tar passes over to the condensers and receivers.

The following figures are taken from a distillation carried out on one of these stills at Provan:

Crude Tar Distillation.

Charge :—32 tons of horizontal tar.
Crude naphtha and water (3%), 22 hours.

Time	12·0	1·0	1·30	2·0	2·30	3·0	3·30	4·0	4·30	5·0	6·0
Temp. at swan neck	191°	222°	230°	240°	262°	273°	286°	300°	306°	306°	325°
Sp. gr. at 60° F.	959	992	1,006	1,013	1,021	1,028	1,035	1,040	1,048	1,060	1,084
Wurtz test.	%	%	%	%	%	%	%	%	%	%	%
120° C.	8	—	—	—	—	—	—	—	—	—	—
130° C.	10	—	—	—	—	—	—	—	—	—	—
140° C.	13	—	—	—	—	—	—	—	—	—	—
150° C.	20	—	—	—	—	—	—	—	—	—	—
160° C.	28	—	—	—	—	—	—	—	—	—	—
170° C.	36	10	3	—	—	—	—	—	—	—	—
180° C.	45	17	13	2	—	—	—	—	—	—	—
190° C.	60	35	20	9	—	—	—	—	—	—	—
200° C.	75	58	45	31	12	8	2	2	2	—	—
210° C.	86	74	60	54	35	25	17	9	6	—	—
220° C.	91	83	76	70	56	45	31	21	13	3	—
230° C.	—	90	84	79	66	60	49	38	23	11	—
240° C.	—	—	89	84	75	70	58	50	34	20	2
250° C.	—	—	—	89	80	77	68	59	43	28	9
260° C.	—	—	—	91	84	82	74	65	53	40	14
270° C.	—	—	—	—	87	86	78	70	59	46	20
280° C.	—	—	—	—	90	90	81	76	64	55	28
290° C.	—	—	—	—	—	—	86	82	70	60	37
300° C.	—	—	—	—	—	—	90	86	76	67	45
310° C.	—	—	—	—	—	—	—	—	80	76	56
320° C.	—	—	—	—	—	—	—	—	83	81	68
330° C.	—	—	—	—	—	—	—	—	87	85	75
340° C.	—	—	salts showing.	more.	very salty.	very salty.	trace salts.	free.	free.	anthr.	85 salts.
Tar acids ..	22	33	32	30	29	27	23	21	15	15	13

* Steam introduced at this point.

(....Light oil....) (....Carbolic oil....) (....Hy. creos.....)

Note.—On the Wurtz test, approximately 85% distils over at the temperature shown on the swan neck at the time the sample was drawn.

Experimental tar distillation.

Egg-ended still. Charge 32 tons of vertical tar.

Time	5 p.m.	6	7	8	9	10	11	12 m.n.	*1 a.m.	1.30	2	2.30	3	3.45 (last runnings)
Temp. at swan neck (° C.) ..	197	220	232	243	253	270	290	302	310	315	320	320	317	310
Sp. gr. of oils collected at coil end	·950	·958	·968	·978	·985	·991	1·001	1·008	1·016	1·011	1·017	1·025	1·025	1·04
	at 15·5° C.									at 30° C.				
Wurtz flask distillation test. ° C.	%	%	%	%	%	%	%	%	%	%	%	%	%	%
120														
130														
140														
150	3	1												
160	7	2												
170		5	3											
180	20	14	8	2	2									
190	39	28	19	8	6	2								
200	60	49	43	27	19	5	3	2	*					
210	76	68	62	51	46	27	13	6	3					
220	88	79	78	66	62	46	30	19	8	3	1			
230	92	87	85	75	73	61	48	36	19	9	5	4	4	
240	96	91	90	82	82	70	61	47	31	20	12	9	6	1
250		93	93	86	87	77	70	57	43	30	24	15	10	3
260			95	89	90	81	75	65	52	41	31	24	15	6
270				91	93	85	80	72	61	50	44	31	24	12
280				93	94	88	85	78	68	59	54	40	31	21
290				94	95	90	88	83	77	65	61	49	38	27
300						92	91	87	80	73	70	58	50	37
310						93	93	90	83	79	80	66	60	47
320							95	93	88	85	83	75	68	57
330							97	96	91	89	88	80	76	66
340								98	93	92	91	85	82	73
350											94	88	87	81
360											97	91	89	86
83% of oil collected below ° C.	216	225	229	244	245	265	277	290	310	317	320	337	342	355
Tar acids per cent. (vol.) ..	30	28	31	38	37	38	30	30	26	20	19	19	19	
	No deposit of "salts."							Oils deposit salts on cooling.						

Time summary.

Tar distilled, 32 tons. Vertical retort tar. Sp. gr. 1·09 at 15·5° C.

From fire lighted to first running	3 hrs.	
From first running to finish of naphtha and water	19¼ hrs.	22¼ hrs.
From finish of naphtha and water to end of distillation		8¼ hrs.
		30½ hrs.

May, 1922.

* Steam applied.

The specific gravity and distillation ranges given, apply to the distillates leaving the condenser coil at the time and temperature stated.

It will be noted that the total time of distillation is 28 hours, and the total distillate is approximately 36-40 per cent. The distillation of the crude naphtha and water, amounting to only 3 per cent., took 22 hours, the remaining 33-37 per cent. coming over in 6 hours. This is a fair average of the time taken for a distillation with this type of still.

It will be noticed that even at 300° C. the distillate still contains 9 per cent. of oils boiling at 210° C.—probably the result of breakdown of more complex organic compounds, which occurs in all distillations.

Fuel used per ton of tar distilled at two of our works, using this type of still.

	For distillation only.	For steam throughout works and in steaming of tar stills.
Works 1.	1·75 cwt.	1·18 cwt.
Fuel used ..	Dross and coke breeze.	Coke breeze.
Works 2.	1·625 cwt.	0·57 cwt.
Fuel used ..	Coke.	Coke breeze.

TYPE 2.

Various types of tar stills, with additional accessories for speeding up the rate of distillation, are in operation. All of these have for their object the preliminary dehydration of the tar by the utilisation of the vapours issuing from the tar stills in a heat interchange system, with consequent saving of fuel.

Fig. 3 illustrates such a system, which is in operation at our Provan Works. It comprises four stills, four preheaters—or heat exchangers—and six condensers. Each still has its separate preheater and condenser. The distillates from each pair of preheaters (crude naphtha and ammonia water) are condensed in one condenser, and all distillate receiving boxes are on one common platform.

The preheater and tar still are charged with tar and the fire lit under the still. The issuing vapours from the still pass through two D-shaped coils in the preheater, imparting some of their heat to the surrounding tar, and are finally condensed by passage through D-shaped coils in the water condenser. At the finish of the distillation the tar in the preheater is practically free from water, and it may be run down into the tar still as soon as the pitch has been run off. The preheater is again charged with crude tar. The dehydrated tar in the still can be fired hard without fear of frothing. We have been able to complete the distillation from firing up to finishing in 6 hours' time.

Summary of observations made during nine consecutive distillations using still with separate preheater.

Averages :—

From charging to charging		7 hrs. 16 mins.
Max.		8 hrs. 42 mins.
Min.		6 hrs.
Charging still, 25 mins.		
Max.		31
Min.		11
Dehydrator, 43 mins.		
Max.		80 (larger charge).
Min.		26
From "Fire on" to "still running" ..		40 mins.
Pitch running, off		19 mins.
Coke used per charge, 10·3 cwt.	Max. ..	15
	Min. ..	8·5
Equivalent to 0·6 cwt. per ton of tar distilled.		
Highest temperature on still		330° C.
On dehydrator		103° C.

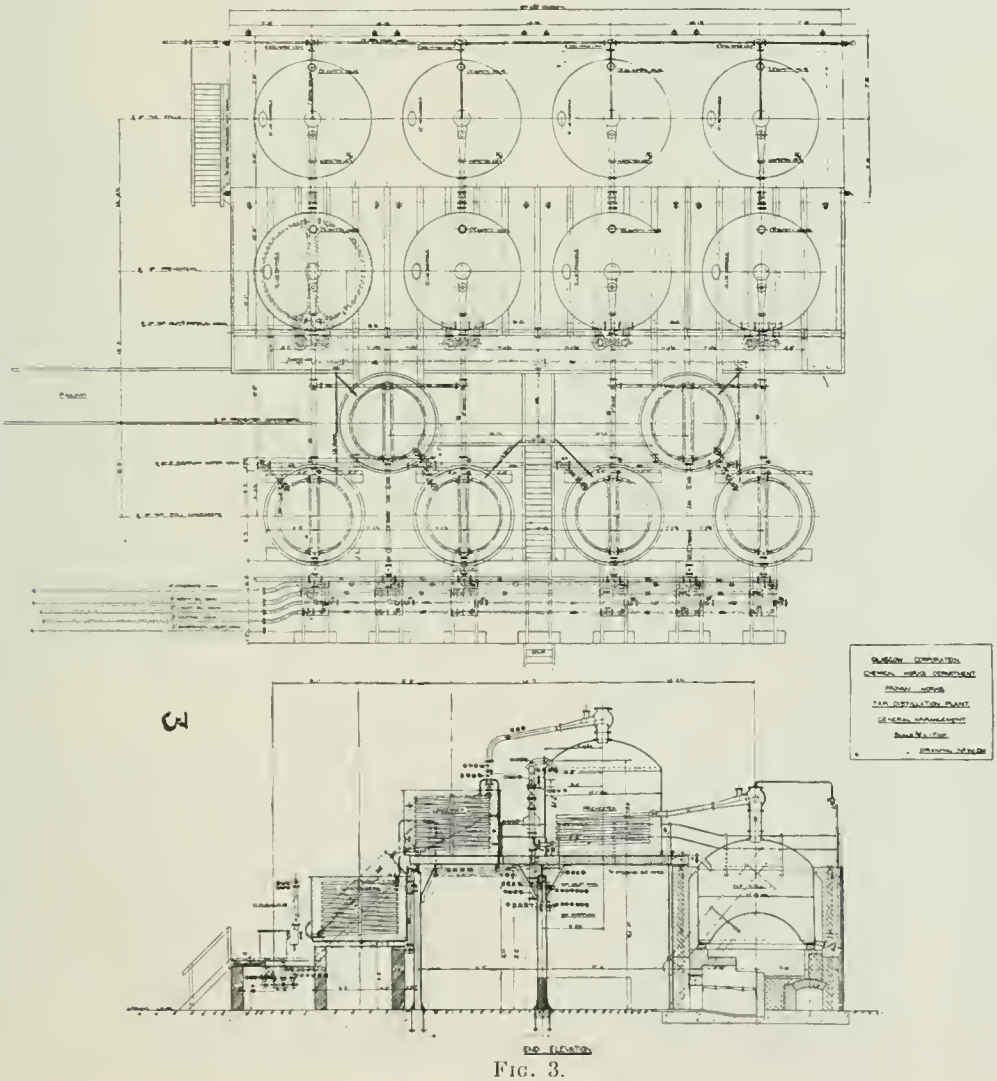


FIG. 3.

The still is 11ft. 0in. diameter with concave bottom, having a 3ft. 0in. dishing. This bottom plate, which is in one piece—so that it can be thoroughly cleaned—is made of $\frac{5}{8}$in. mild steel plate. The shell is made in two halves riveted, and the dome in two halves welded. It will be noticed that all pipes connected with the above the pitch outlet pipe, and for a distance of 9in. on each side of the centre line, the space is filled in with a 4½in. brick wall. By removing this, the still can be lifted bodily from its setting, and a new still dropped into position, thus obviating the taking down of the setting to replace stills.

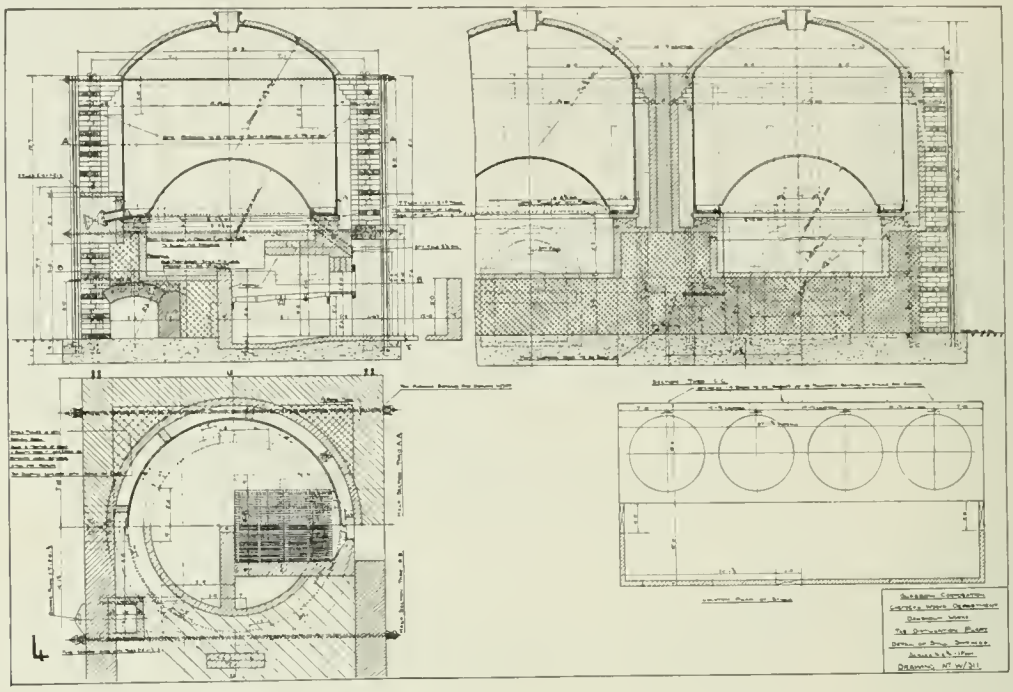


FIG. 4.

dome project through inside the still. This is to ensure that in case any condensation takes place on these pipes, it should drop clear into the tar below. If this is not done, serious corrosion in the dome and sides of the still may take place through condensation of dissociated ammonia compounds.

The swan neck or still head is of special type, with a circular upstand to prevent any condensation at this point running back to the still. Fig. 3 shows plainly the preheater and condenser coils with breeches pipes connecting same with other portions of the vapour oil pipe-line. Each D-shape coil is in one piece lapwelded. The D-shaped coils for the preheaters are 3in. diameter, giving a total heating surface per coil of 237 sq. ft. The condenser coils are 2in. diameter, giving a total cooling surface of 200 sq. ft.

The tar still setting has several points worthy of mention, and is shown in Fig. 4.

The seating consists of 9in. circular brick wall with pigeon holes, on the top of which are laid fire tiles, 18in. square × 3in. deep, to protect the still bottom where the hot gases pass through the pigeon holes to the outer flue. This outer flue extends practically to the top of the straight portion of the still, and is divided by mid-feather walls, built between the pigeon holes, and running to about two-thirds the height of the flue. This ensures that the gases passing through the pigeon holes pass straight up the side of the still before gathering together at the top and cutting round to the outlet connected to the chimney stack, thereby ensuring even wear of the sides of the still. From the top of the still setting to just

TYPE 3.

The Wilton tar dehydration coil, in conjunction with pot stills.

This type is in operation at the Corporation's Dalmarnock Works. The first unit, consisting of one dehydration coil, and two pot stills, has recently been duplicated.

Fig. 5 shows, in plan and section, the naphtha stripping plant at Provan, and as both plants are practically the same, one description will serve for both.

(A) "Wilton" furnace for coke breeze.
(B) Centre gas flue.
(C) 3in. cast iron coil surrounding B.
(D) Flue gases to chimney.
(G) Vapour chamber in sections, with bubbling trays and inlet for steam, having vapour outlet, H, and residual oil outlet, J.
(K) Condenser.
(M) Heat exchangers.
(N) Oil pump.
(O) Inlet pipe to coil through heat exchangers.
(P) Outlet pipe from coil to vapour chamber, G, with pressure gauge, thermometer, and cock to regulate pressure.

The furnace gases from A pass up B, and down over coil, C, to chimney, by route D. The oil is pumped by N through the heat exchangers and through the coil, leaving at temperature of about 160° C., under a pressure of 30 to 40lb. per sq. in. It is then released to atmospheric pressure in vapour chamber, G., naphtha vapour passing over by H to condenser, K. The residual oil passes from tray to tray in G, meeting an upward current of steam if this is necessary to eliminate

more naphthas, and finally emerging by pipe, J, to pass through heat exchangers, M, where it heats the ingoing oil and is itself cooled.

In the case of tar dehydration, the only difference is that there are no heat exchangers—the vapour chamber, G, is just an open box with no trays and the residual dehydrated tar passes tillation plant, to produce pitch of merchantable quality.

The crude tar was dehydrated in the usual way in the first coil. It was then pumped through the second coil (which was maintained at considerably higher temperature, but at a slightly lower pressure than No. 1) and released into the

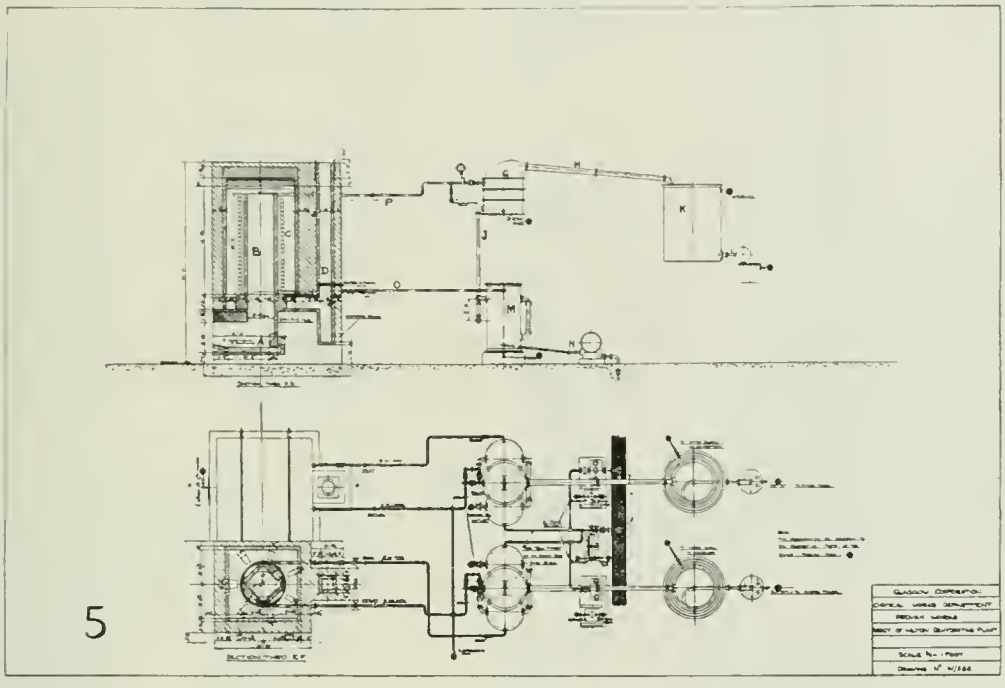


Fig. 5.

from J into the tar still. Each dehydration coil is capable of dehydrating 10,000 gallons of crude tar per day, or of stripping the naphthas from 10,000 gallons of oil per day.

CONTINUOUS TAR DISTILLATION EXPERIMENT AT DALMARNOCK WORKS.

Tar distilled—all vertical retort tar.

The object of the experiment was to see if it were possible to run two "Wilton" dehydration coils with a small final steaming still of about 500 gallons capacity, as a continuous tar dis-second vapour chamber. The residual tar then ran by gravity into the final steaming still (which was kept hot by waste furnace gases from coil No. 1) where it was subjected to steam distillation with superheated steam. The outlet from the steaming still could be raised or lowered to give longer or shorter time for steam distillation as might be required, whilst the thick tar was passing through continuously to the pitch coolers.

In the first experiment the temperature of the tar leaving the second coil was maintained at 280° C. at a pressure of 25 to 30lb. per square inch, and a very soft pitch was obtained. The temperature was then raised to 300° C. and then to 320° C. at the same pressure; the resulting

CONTINUOUS DISTILLATION EXPERIMENT.

Date.	Time.	Tar distilled.	Crude Naphtha and water.	Middle oil.	Heavy oil.
	3.15 p.m.	Zero. Galls.	Zero. Galls.	Zero. Galls.	Zero. Galls.
April 3rd, 1922	6.25 p.m.	1,160	63	629·5	10
April 4th, 1922	8.0 a.m.	4,827	582	2,994	235
,, ,, ...	12.30 p.m.	6,274	750	3,789·5	252
,, ,, ...	3.45 p.m.	7,184	870	4,454	332
,, ,, ...	6.0 p.m.	8,809	931	5,025	332
,, ,, ...	12.0 night	10,801	1,005·6	7,407	—
April 5th, 1922	4.0 a.m.	12,226	1,197	8,361	—
,, ,, ...	8.0 a.m.	13,762	1,437	9,069	—
,, ,, ...	11.05 a.m.	14,897	—	9,742·3	525
,, ,, ...	12.0 noon	15,289	1,595	9,943·5	525
,, ,, ...	5.30 p.m.	17,199	2,135·7	11,278·5	585
,, ,, ...	8.25 p.m.	18,159	2,725·1	11,860	—
,, ,, ...	12.0 night	19,999	3,330	12,556·8	722
April 6th, 1922	3.30 a.m.	20,757	3,706·3	13,250·5	—
,, ,, ...	8.0 a.m.	22,743	4.041·9	14,277·4	822·3
,, ,, ...	12.0 noon	23,976	4,541·3	15,038·2	—
,, ,, ...	6.50 p.m.	26,992	5,679·3	16,338	830
,, ,, ...	8.45 p.m.	27,904	6,064	16,871	834·3
,, ,, ...	12.0 night	28,954	6,622·2	17,617·9	871
April 7th, 1922	5.15 a.m.	30,404	7,181·9	18,796·5	871
,, ,, ...	8.0 a.m.	31,892	7,330·0	19,423	902·7

Date.	Time.	No. 1 Coil. Temp.	No. 1 Coil. Pressure, lb. per sq. in. Entrance, lb.	No. 1 Coil. Pressure, lb. per sq. in. Exit, lb.	No. 2 Coil. Temp.	No. 2 Coil. Pressure, lb. per sq. in. Entrance, lb.	No. 2 Coil. Pressure, lb. per sq. in. Exit, lb.	Temp. of tar leaving 2nd vapour chamber.	Finishing still exit temp.	Twist point of pitch.
April 3rd, 1922	3.30 p.m.	150° C.	32	38	350° C.	27	22	250° C.	210° C.	—
,, ,,	4.0 p.m.	182° C.	32	37	347° C.	24	20	254° C.	220° C.	—
,, ,,	4.30 p.m.	180° C.	29	29	358° C.	24	20	245° C.	230° C.	—
,, ,,	5.0 p.m.	147° C.	30	32	320° C.	26	26	282° C.	218° C.	—
,, ,,	5.30 p.m.	215° C.	40	40	320° C.	30	26	260° C.	233° C.	—
,, ,,	6.0 p.m.	185° C.	38	38	342° C.	30	18	245° C.	225° C.	—
,, ,,	6.30 p.m.	190° C.	44	41	375° C.	26	23	233° C.	224° C.	119° F.
,, ,,	7.0 p.m.	170° C.	26	25	350° C.	24	21	269° C.	234° C.	120° F.
,, ,,	11.20 p.m.	160° C.	20	18	345° C.	21	18	—	—	104° F.
April 4th, 1922	4.45 a.m.	165° C.	30	27	340° C.	28	24	—	—	115° F. (2 a.m. 108)
,, ,,	8.0 a.m.	145° C.	35	30	360° C.	24	22	—	—	110° F.
,, ,,	9.30 a.m.	177° C.	33	35	348° C.	24	25	234° C.	195° C.	—
,, ,,	10.30 a.m.	180° C.	33	30	354° C.	26	22	265° C.	225° C.	125° F.
,, ,,	11.30 a.m.	175° C.	30	30	345° C.	30	26	252° C.	230° C.	112° F. (1.30 p.m. 114)
,, ,,	2.30 p.m.	200° C.	27	28	355° C.	32	29	270° C.	233° C.	126° F.
,, ,,	3.0 p.m.	198° C.	24	24	342° C.	26	24	225° C.	205° C.	—
,, ,,	4.30 p.m.	145° C.	29	32	350° C.	24	20	258° C.	231° C.	—
,, ,,	5.0 p.m.	160° C.	29	28	350° C.	24	20	234° C.	225° C.	—
,, ,,	6.0 p.m.	195° C.	30	28	350° C.	24	21	227° C.	214° C.	126° F.
,, ,,	8.0 p.m.	195° C.	30	29	347° C.	24	19	260° C.	229° C.	—
,, ,,	10.0 p.m.	205° C.	30	30	342° C.	25	22	271° C.	242° C.	128° F.
,, ,,	12.0 night	195° C.	32	33	360° C.	24	22	297° C.	235° C.	130° F.
April 5th, 1922	2.0 a.m.	180° C.	30	31	350° C.	25	21	292° C.	278° C.	140° F.
,, ,,	4.0 a.m.	175° C.	25	27	349° C.	22	18	295° C.	245° C.	135° F.
,, ,,	6.0 a.m.	170° C.	27	26	352° C.	23	20	266° C.	250° C.	125° F.
,, ,,	8.0 a.m.	175° C.	28	26	350° C.	24	19	262° C.	226° C.	131° F.
,, ,,	10.0 a.m.	192° C.	24	24	360° C.	23	18	247° C.	—	—
,, ,,	11.0 a.m.	170° C.	34	35	356° C.	20	20	237° C.	—	132° F. (1.45 p.m. 131)
,, ,,	2.30 p.m.	180° C.	32	37	350° C.	24	24	290° C.	245° C.	142° F.
,, ,,	4.30 p.m.	180° C.	38	38	358° C.	23	20	260° C.	240° C.	145° F.
,, ,,	7.30 p.m.	175° C.	40	38	340° C.	24	20	285° C.	270° C.	145° F.
,, ,,	8.0 p.m.	175° C.	42	40	350° C.	23	19	—	—	—
,, ,,	10.0 p.m.	190° C.	34	36	360° C.	22	18	255° C.	239° C.	125° F.
,, ,,	12.0 night	170° C.	36	36	340° C.	24	18	270° C.	263° C.	124° F.
April 6th, 1922	2.0 a.m.	175° C.	33	30	345° C.	22	18	272° C.	238° C.	127° F.
,, ,,	4.0 a.m.	140° C.	32	30	340° C.	24	20	262° C.	250° C.	127° F.
,, ,,	6.0 a.m.	160° C.	34	34	340° C.	23	18	287° C.	236° C.	125° F.
,, ,,	8.0 a.m.	170° C.	38	39	340° C.	23	18	230° C.	212° C.	126° F.
,, ,,	10.0 a.m.	160° C.	40	40	351° C.	23	18	290° C.	220° C.	—
,, ,,	10.25 a.m.	—	—	—	360° C.	—	—	—	—	131° F. Superheater = 410° F. Boiler Press 95 lbs. (Steam shut off finishing still.)
,, ,,	2.0 p.m.	170° C.	38	38	330° C.	20	12	244° C.	215° C.	126° F.
,, ,,	3.0 p.m.	165° C.	29	25	338° C.	20	16	270° C.	263° C.	130° F.
,, ,,	5.0 p.m.	170° C.	36	33	340° C.	24	19	258° C.	253° C.	130° F.
,, ,,	8.0 p.m.	180° C.	35	35	350° C.	28	23	—	265° C.	114° F.
,, ,,	10.0 p.m.	185° C.	36	35	355° C.	28	23	—	—	120° F.
,, ,,	12.0 night	170° C.	45	45	345° C.	27	21	—	250° C.	127° F.
April 7th, 1922	2.0 a.m.	175° C.	39	38	348° C.	25	18	—	265° C.	134° F.
,, ,,	4.0 a.m.	124° C.	36	32	327° C.	22	16	—	262° C.	123° F.
,, ,,	6.0 a.m.	155° C.	29	26	335° C.	21	15	—	264° C.	122° F.
,, ,,	8.0 a.m.	165° C.	39	36	342° C.	25	20	—	237° C.	123° F.

* Wood's Alloy M.P. 73° C. was used in the thermometer cups. Mercury was found unsuitable for the higher temperatures.

Total tar distilled	32,660 galls. = 158 tons
Duration of test	88¾ hours
Fuel used at No. 1 coil	9 tons
,, ,, No. 2 ,, ...	10 tons 12 cwt. 2 qrs.
Total fuel used	19 tons 12 cwt. 2 qrs.
Fuel used per ton of tar distilled	2·48 cwt.
,, ,, 1,000 galls. on No. 1 coil	617 lbs.
,, ,, ,, No. 2 ,, ...	730 ,,

Rate of working :—

1st	24 hours ..	330 galls. per hour	(10,000 galls. per 24 hours).
2nd	24 ,, ..	380 ,, ,,	(=416 galls. per hour).
3rd	24 ,, ..	370 ,, ,,	
Last	12 ,, ..	245 ,, ,,	

pitch twisted at 80—90° F. The temperature was finally raised from 350° to 360° C., and a good medium soft pitch twisting at 124° F. was obtained.

No difficulty whatever was experienced, and the object of the experiment having been accomplished, interest shifted to the possibilities of cracking, as larger volumes of oil than usual had been obtained along with a reduced quantity of pitch.

It has been pointed out in the introduction to the paper that vertical-retort tar is really a mixture of high- and low-temperature tars, the low-temperature portion being essentially paraffinoid in nature, the phenolic compounds of complex composition containing polyhydric phenols with possibly paraffinoid side chain linkings.

Under the conditions of the experiment, the breakdown of these complex phenolic compounds was thought to be highly probable, and gave rise to interesting speculations as to whether breakdown to simpler phenols and paraffins would take place, or possibly polymerisation with formation of resinous substances.

A second experiment, over a longer period, was undertaken in order to confirm the results obtained in the first experiment. Full details of the second running are appended. Unfortunately, owing to shortage of tar, the experiment had to be stopped at the end of the fourth day.

Some time during the night on April 6th, we came on to very watery tar, containing up to about 30 per cent. of water. The results have therefore been summarised up to 6.50 p.m. on the 6th, up to which time tar containing a normal percentage of water had been used:—

Tar distilled up to 6.50 p.m. 6/4/22—
22,664 gallons dry tar.

Oils made :—

	Galls.	Sp. gr.	
Crude naphtha ..	1,351 at 50° C.	0·905 = 5·46 tons	
Middle oil	16,338 at 60° C.	0·970 = 70·7	,,
Heavy oil	830 at 20° C.	1·043 = 3·88	,,
		80·04	,, = 73%

Pitch and loss by difference = 27% of tar distilled.

In the normal course of working, the yields of oil and pitch are respectively 56-58 per cent. of oils, and 41-43 per cent. of pitch.

It will be noticed that very little work was done in the final steaming still. It was observed, however, that when the temperature of the tar leaving the second coil fell, more work was done by this final steaming still.

It will be seen from the result that it was not necessary to use the steaming still at all to obtain a good medium soft pitch.

Since the experiment, the top portion of the second coil has been dismantled, and six laps show a deposit of carbon. The first lap shows from $\frac{3}{8}$" to $\frac{1}{2}$" deposit. The next three show a $\frac{1}{4}$" deposit, and the next two $\frac{1}{8}$" deposit of carbon.

The oil produced from the second coil during the experiment has been examined, and a comparison is given below between this oil and the oil obtained by distillation in the pot still.

	A. (Oil obtained during experiment.)	B. (Mixed carbolic and heavy creosote oils obtained during ordinary distillation in pot stills.)
Sp. gr. 15·5° C.	1·03 ·991 (Fraction to 300° C.). ·9568 (Fraction to 300° C., washed free from tar acids and pyridine).	1·014 ·987 (Fraction to 300° C.) ·9547 (similarly treated).
Paraffins ..	15 (Fraction to 300° C.).	12 (Fraction to 300° C.).
Loss on washing with H_2SO_4, 1·84 sp. gr.	(16Fraction to 300° C.). Washed free from tar acids and pyridine.	12 (Similarly treated).
Tar acids ..	34%	34%
Sp. gr. crude dry tar acids	1·087	1·083
Character of tar acids ..	Apparently identical	
Calorific value	17,100 B.T.U.	17,400 B.T.U.
Residual at 300° C.	Stiff sticky homogeneous mass. No evidence of low boiling bodies.	Mobile oily mass containing crystals.

From these figures it would appear that there is very little difference between the fraction of either "A" or "B" boiling up to 300° C.

On carrying the distillation of both oils to 360° C., oil "A" leaves a residue of soft pitch, amounting to 24%, against 9% from oil "B."

Examined microscopically the pitch produced during the experiment appeared to contain no more carbon than that produced in the ordinary method of distillation by the pot still.

CARBOLIC PLANT.

Oil Washers.

These are of the Typhoon type, illustrated in Fig. 6. This is a very efficient type of washer.

The washer having been charged with oil and caustic soda solution, the paddle, A, is set in motion, the quickly revolving contents setting up a vortex in the centre. The radial baffles, B, are then lowered so as just to break the surface of the liquid, which is thus caused to cascade into the centre of the vortex. When agitation is complete and just prior to settling, if the radial baffles are raised clear of the liquid and the propeller kept in motion for a short time, quicker separation takes place. The lower layer of sodium salts of tar acids is run off and pumped into the carbonators. The carbonators are old tar stills. When fully charged, carbon dioxide obtained from waste gases from the sulphate of ammonia plant is bubbled through, liberating the tar acids and forming sodium carbonate.

Dorr Continuous Caustieising Plant.

Figure 7—Flow Sheet.

The lime and sodium carbonate are slaked in a Harding conical ball mill. The slaked lime etc. flows into a sump, and is pumped to the first reaction agitator, overflowing from there to the second reaction agitator, and from the second

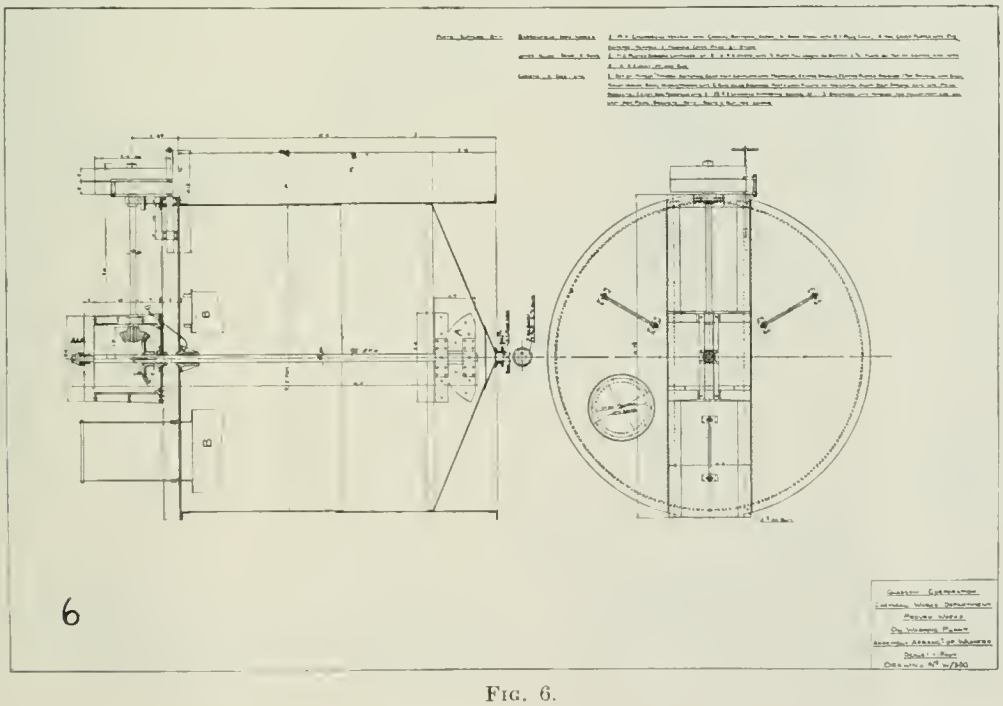
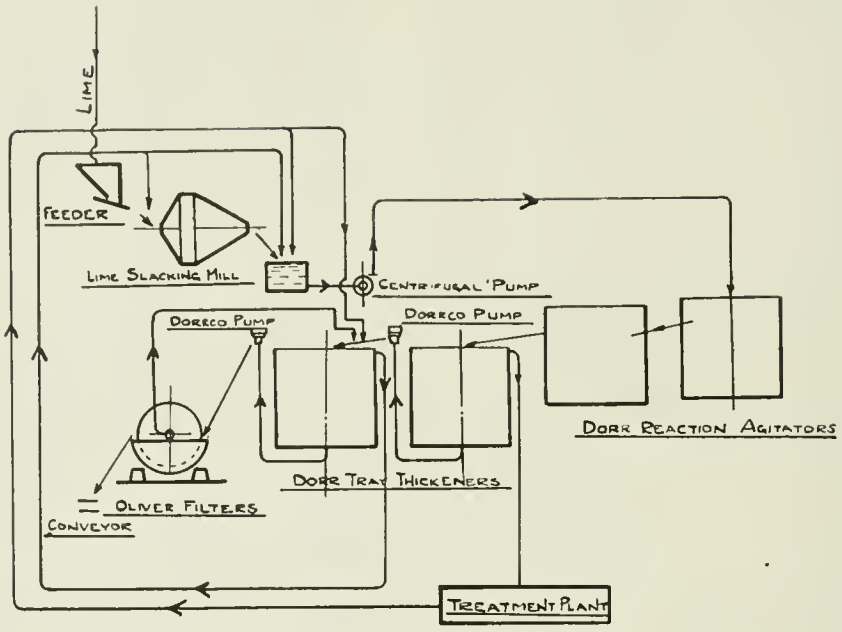


Fig. 6.



7

FLOW SHEET
OF
DORR CONTINUOUS CAUSTICIZING PLANT

FIG. 7.

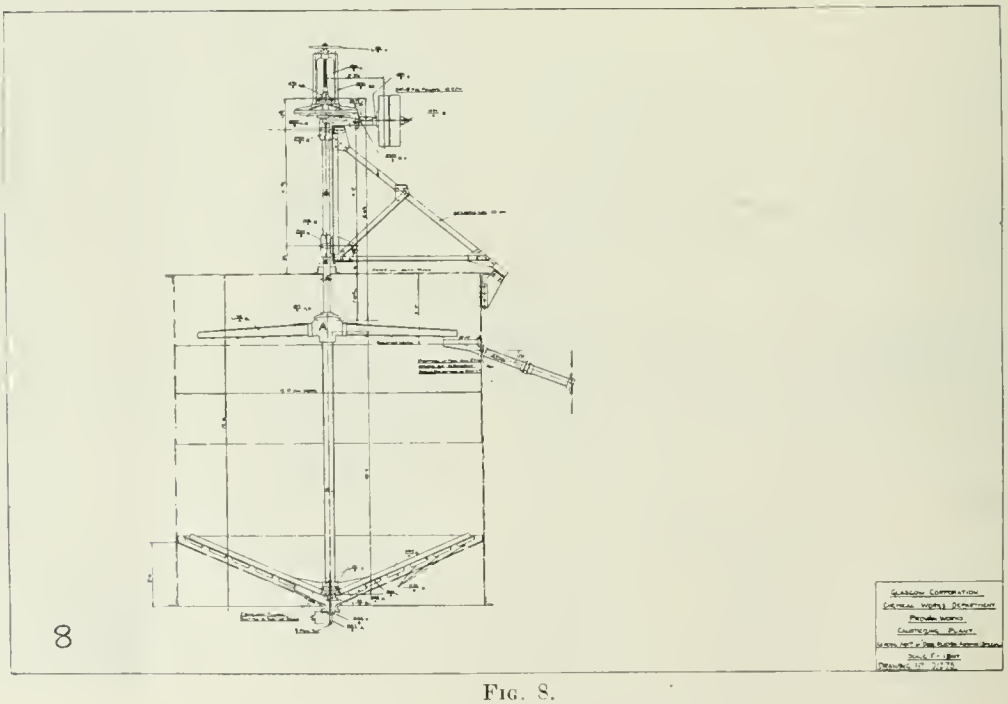


FIG. 8.

to the Dorr thickener. Caustic soda solution (approx. 10 per cent. NaOH) overflows near the top of the thickener, and the underflow of calcium carbonate sludge is pumped to the next thickener, where it meets wash water as it emerges

"Dorr" reaction agitator. (Fig. 8.)

The drawing is self-explanatory. It is only necessary to point out that the shaft below A is hollow, and has an inlet for compressed air

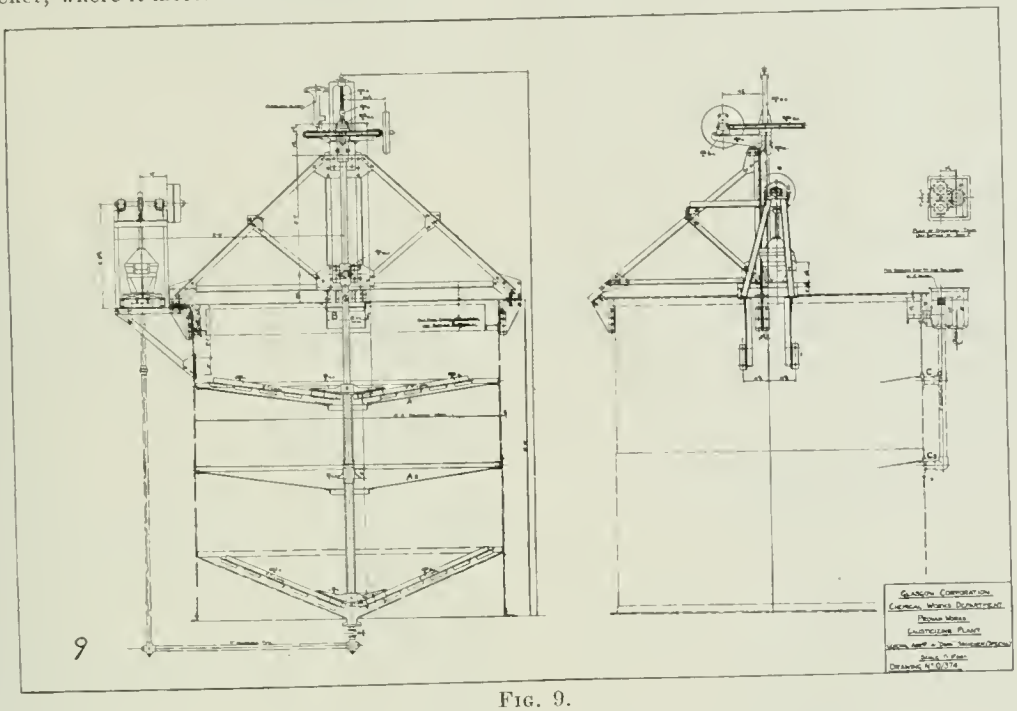


Fig. 9.

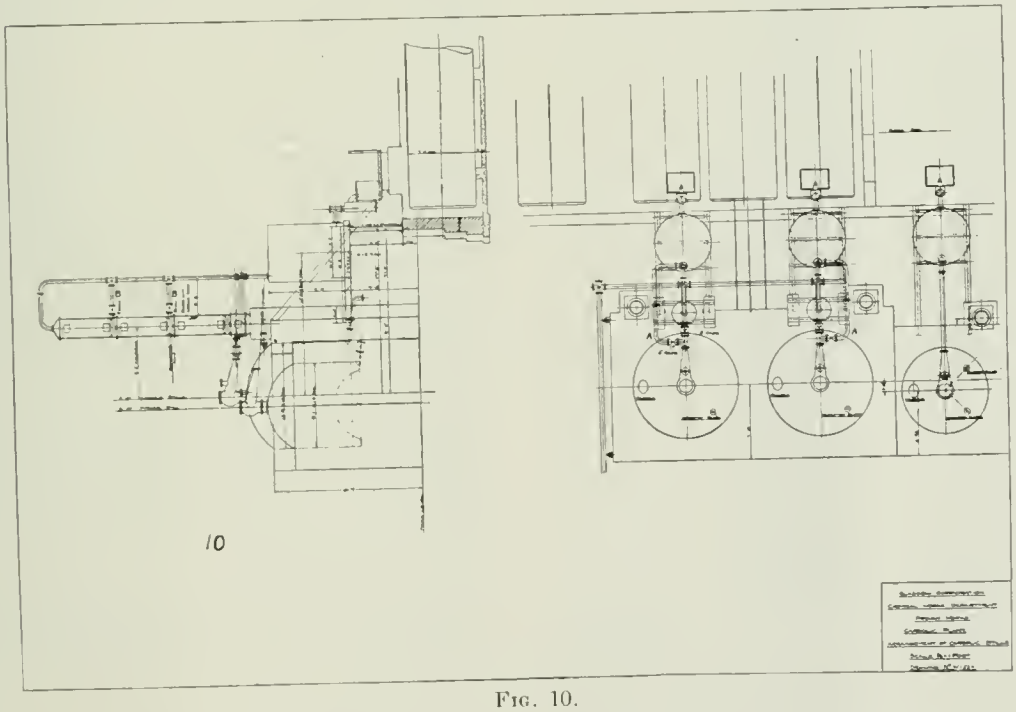


Fig. 10.

from the pump. Weak caustic soda overflows, and is returned into process, and the underflow of calcium carbonate is pumped to the Oliver filter. The filtrate from the Oliver filter is returned into process, the calcium carbonate cake being removed by conveyor.

at the bottom. The compressed air ejects the reacting mixture up the hollow shaft on to A which is perforated on the bottom, and acts as a sprinkler. The reaction agitators are lagged to conserve heat, and additional heat is obtained by means of steam coils, not shown in the drawing.

"Dorr" thickener. (Fig. 9.)

The "Dorr" thickener is divided into three sections by the trays, A and A_2. It has three sets of paddle arms (the arms on A_2 are not shown). The overflow from the second reaction agitator enters the thickener at B. The paddles revolve very slowly. It has been shown by the "Dorr" people that quicker settling of suspended matter from a liquid is obtained by gentle agitation, than by allowing settling to take place in the ordinary way.

Clear caustic soda solution flows away by pipes C and C_2, just under the outer edge of trays, A and A_2.

The thickeners are lagged to prevent convection currents being set up by external cooling, which would impede settling.

The Oliver continuous rotary filter, and the Scott evaporator, have, I believe, been described at previous meetings of this Group, and require no comment.

It was hoped that the plant would be in operation in time for the Meetings, but this does not now appear possible.

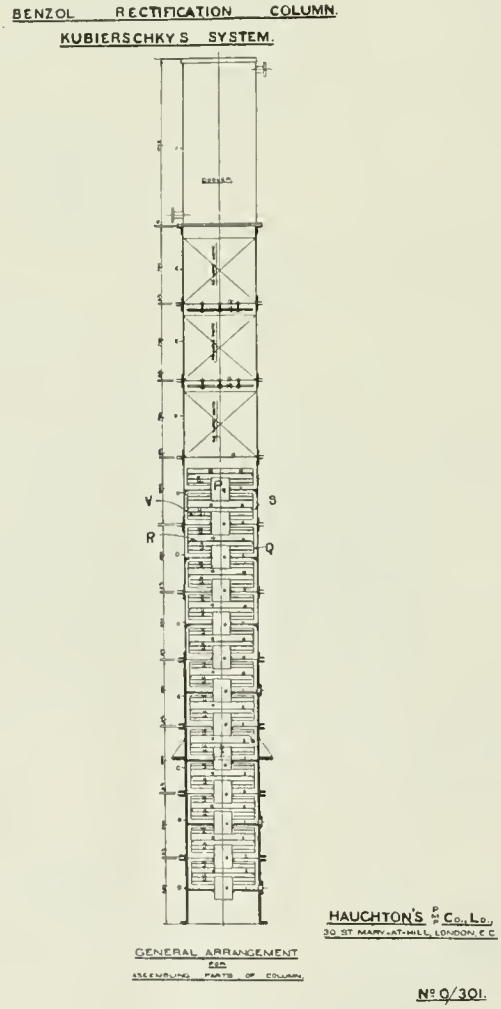


Fig. 11.

Carbolic Distillation Plant.

Fig. 10 illustrates this plant with fractionating column in 6ft. sections, packed with Lessing rings. Until the distillates are free from water, all vapours bypass the column by pipe, A, to the condenser. Vapours passing up the column may be taken off through pipes, B, B, at the top of the first and second sections, if it is not desired to use the whole column at any period during the distillation. The rings are dumped into the column and allowed to arrange themselves in whatever position they fall. Only 10-20 per cent. of the volume of the column is taken up by the material of the rings. The column, therefore, has a large amount of free space, and offers little resistance to the vapours.

Benzol plant.

This plant contains two Hodson Thomas stills with columns and dephlegmators and one still with Kubierschky spraying column with dephlegmator.

The Hodson Thomas stills are heated by a nest of tubes fitted into expansion tube plates at each end of the still. The steam chest is divided longitudinally by a plate which is part of the steam chest casting and each half has its own steam connexion, so that half or all of the tubes can be utilised as desired.

The column is of the bubbler type and is built up of a number of cast iron sections. A loose plate carrying four serrated caps fits into each section. The return pipe from section to section is arranged to give a sufficient depth of liquid on each tray to form a seal.

The dephlegmator is of the usual type, tubes expanded into expansion tube plates. The vapours enter at the top, cooling water passes up the tubes, and any condensate passes back to the column by means of a return pipe from the bottom.

The uncondensed vapours, *i.e.*, the lighter fractions, pass forward to the condenser.

Kubierschky spraying column.

The column for two-thirds of its height, is made up of a number of sections. Each section has centre vapour passages, P, surrounded by cylindrical casings forming with the shell of the column annular spaces, S. The cylindrical casings, Q, are divided into sections by the perforated plate, R (this plate is not perforated in the centre above the vapour passage, P), and the stabbed plates, V. The top portion of the column is filled with wire mats.

The vapours rising through P descend with the downward raining condensates and are forced by the vapours following to the bottom of the compartment and up through the annular space, S, to the next top of the next compartment. The vapours pass in the same way to the bottom and rise up through the next centre vapour passage and so on, in the directions indicated by the arrows.

It is hoped that some useful information may be obtained from the paper. One regrets that it is not possible to give any figures in reference, particularly to the continuous causticising plant and Oliver filter, or to compare from actual working the relative merits of the three types of rectification columns described, as the plant is not yet completed.

In conclusion, I wish to express my thanks to Mr. R. F. Stewart, for assistance in carrying out tests and compiling data relative to tar acids and the continuous distillation of tar, and to Mr. C. Chapman for assistance in compiling other data embodied in the paper.

Discussion.

The Chairman said he was sure they had all listened with very great interest to this paper, and perhaps later on he would have something to say upon it. For the moment, all he would do was to draw attention to the very pretty way in which the plant had been arranged. He was sure that must have impressed itself upon all those who had had the opportunity of visiting the works the previous day.

Mr. F. Nicholls asked the author if he dealt with his horizontal and vertical retort tar separately or together, and what advantage did he find from whichever course he adopted?

Mr. R. McLauren said he wished to point out the necessity for a much better nomenclature in regard to different tars. It was not sufficient now to discuss tars as coal tars. There were high temperature tars and low temperature tars, but even that was not sufficient, and it would be necessary to define tar as high temperature tar, semi-high temperature tar, medium tar, semi-low temperature tar and low temperature tar. There were very big differences and the paper indicated some of them. The ordinary horizontal tar was rightly described as high temperature tar. Vertical tar was deseribed by the author as a mixed tar, which he also thought was correct, although in the main it was really a medium temperature tar. The great number of tars that were being produced just now by low temperature processes were not truly the lowest temperature tars; they were what he would call semi-low temperature tars. The only true low temperature tars that he knew were those that were carried forward immediately out of the temperature at which they were distilled. In most low temperature processes, where there was external heating, the gases reached a temperature of 500° or 600° and decomposition rapidly set in. That decomposition set in with a true low temperature tar. If they put true low temperature tar into a still and distilled it, a very large amount of dissociation took place under these conditions. That was the necessity he saw for a further definition of high and low temperature tars. The next point in the paper to which he wished to draw attention was where the author referred to the possibility of the production of low temperature tar in the ordinary vertical retort, and to some solvent action on the coal at these lower temperature zones so that the gases picked up, in passing, some of the primary constituents of the coal stuff itself. He did not find that in the McLauren process, and he was inclined to think it was not so. The author also mentioned the probability of the presence of trihydric phenols in the higher boiling portions in connection with vertical retorts. In low temperature tar there certainly was a body very similar to pyrogallol, although he had not been able to locate it. It was present in considerable quantity and was like pyrogallol, but he could not say definitely whether it was a trihydric phenol. A very interesting point with regard to horizontal and vertical retort tar was the percentage of tar acids compared with the percentages of tar acids in low temperature tars. It was really a question of distillation. According to the paper there was 9% of crude tar acids from a high temperature tar, and that worked out, ronghly, at 9 lb. per ton of coal. If they took vertical retort tar there was a higher yield of tar acid per ton, viz., about 20 lb., whereas with the true low temperature tar the proportion was 40 lb. per ton. The characteristic of the low temperature tar was the higher percentage of the higher boiling point phenols—and that was further evidenced in the paper, where there was an indication of a rapid decline in the high boiling point phenols in vertical retort tars, whereas the percentage of high boiling point phenols in low temperature tars was of the order of 30% to 40%. Even at 250° to 300° there were still phenolic bodies in the pitch. He wished to suggest that these high boiling point phenols were being decomposed in the ordinary process of distillation and gave benzene. Was it possible that naphthalene occurred in high temperature distillation just at the point of distillation of the higher boiling point phenols? He thought it worth while considering that, because the naphthalene appeared as the high boiling point phenols disappeared. It did not follow, because there were other aromatic bodies in the tars, but it seemed to him a suggestion worth following up.

Mr. P. Parrish said it seemed to him that the setting which the author had adopted so far as the newer type of still was concerned only gave a short time contact as between the products of combustion and the still by reason of the fact that he adopted a vertical flue. He knew that one of the considerations in connection with that arrangement was the fact that the still could be lifted out and there was no necessity to replace the brickwork, but it seemed to him that that advantage could be obtained and yet a design of flue adopted which would admit of ten times the contact possible with the ordinary ones. He wondered if the author had considered it, because he had occasion during the war to seat many stills, and found that from the point of view of efficiency the spiral type of flue was infinitely better. With regard to the Typhoon agitator, it seemed to him that there was an optimum agitation possible with that type and that when they got beyond it emulsions were formed. He wondered whether the author had had any experience in that connection. His third point was the question of the Kubierschky column, the bubbling type of columns and Lessing rings. It was highly important that chemical engineers should arrive at some coefficient with regard to these various fractionating columns. This work ought to have been done long ago, but Mr. Walmsley had an admirable opportunity for trying their respective merits and seeing precisely which was the best. He had had many discussions with Dr. Lessing on this subject, but he—Mr. Parrish—still believed in bubbling. He had had a good deal of experience in connection with bubbling as compared with time contact, and had yet to be convinced that the enormous areas that had to be afforded in connection with time contact in order to admit of fractional condensation were at all comparable with the small column and high efficiency which resulted from bubbling. Therefore, he suggested that Mr. Walmsley had an admirable opportunity for determining once for all what the respective coefficients were, and the chemical industry generally would be grateful to the author if he would publish the data at some future time.

Mr. F. M. Potter said the best contribution that he himself could make to the discussion would be to give some figures for the working of Wilton coils, of which he had had some considerable experience. He greatly favoured the system of a Wilton coil combined with the pot still, using one Wilton coil very much on the lines laid down in the paper, and using a pot still for taking off the light, medium and heavy creosote fractions. The advantage of this system was that they could deal with any tar no matter how bad it was, and the process was almost independent, so far as frothing troubles were concerned, of the ammoniacal liquor content. The only trouble as the water increased was a reduction in output and an increased repairs charge on the stills due to the increased ammonium salts present. He had erected units to deal with 30,000 gallons in the 24 hours, in addition to units

similar to those mentioned in the paper. The 30,000-gallon units, however, were not built as a vertical coil but as a horizontal coil in two layers, the top layer being, as it were, a preheating section. With a coil of that kind he had found it possible to get a fuel figure per 1000 gallons of tar of 240 lb. of comparatively low grade coke breeze, which would run out at about 10,000 B.Th.U. per lb. The fuel used on the pot still would be about 340 lb., making a total of 580 lb. of fuel per 1000 gallons of tar, that tar having an average of 8% to 10% of water. These figures could be compared with those given in the paper. His doubts with regard to the double coil system were partly as to repairs and maintenance of the second coil. He believed that in the earlier days with this coil a somewhat similar system was tried and was not found to be very successful. In the present case he knew that the initial life of the coil, without any repairs whatsoever, would be between 10 and 11 million gallons of tar at the first run. After that, the bottom layer commenced to go, and there would be a breakdown after another 1½ to 2 million gallons. These repairs, however, were easily effected. It meant the removal of one or two 9 ft. lengths of pipe or bends, and replacement of a small section of brickwork. The life between repairs got shorter each time until a figure of 15 or 16 million gallons had been reached. Then it was best to remove the bottom layer of the coil and replace it and thus start again with a fresh initial life for this section. With this combined system, he got very steady working on the pot stills. The 30,000 gallon coil kept four stills going and employed two men per shift, one working the coil and one working the stills, so that they had two men per shift per 30,000 gallons of tar in the 24 hours. That meant that the still was running about three charges in the 24 hours on the 12½ ton still, which means quite swift working. The average workings in his case for the 12 months was just over 100 charges per still between cleanings. He believed that these figures would be interesting when they were considering the working of the double Wilton coil. The advantages of the double coil were still open for further discussion. If they were going to get the cracking, which the author referred to, that was a point which would have to be considered against the advantages of the other plant. He would like to suggest to the author that in going into this question of cracking, he should endeavour to trace its extent by getting out a free carbon balance as between the free carbon in the original tar and the free carbon in the final pitch. He had sometimes suspected that there might be more cracking in the Wilton coil as compared with the pot still on his small experimental unit and he had endeavoured to trace it by determining the free carbon in the tar fed in, and the free carbon in the final product and estimating the formation of free carbon by difference, but he found that the amount formed was exceedingly small. He had no figures from other works, but there were some American patents in which the patentees claimed to effect some great improvements in tar distillation by conducting it in an atmosphere of CO_2. They actually bubbled CO_2 or nitrogen gas through the still, and in the preamble to one of the patents they claimed some very large improvements in the amount of free carbon formed during distillation.

The Chairman said he was not absolutely sure of his ground, but he believed he was right in saying that the inventor of the pre-heater system had been rather forgotten in the paper. He believed that the first patent for the pre-heater system was taken out way back in the last century by Mr. Ellison and he deserved the credit because it was not taken up for a long time afterwards. He felt that that ought to be mentioned. He did not know that he had very much to say beyond that except that he had got some figures that might be of interest. Some years ago he went into the question of pre-heater stills and had some large scale experiments carried out to try and find out how much heat was available, and the results were interesting. Four experiments were carried out. In the first case there was an interval of a month, after the first two experiments had been carried out in the same week, and then there was an interval of another month between the third and the fourth experiments, so that weather conditions came in. He measured the water into the condenser and measured the rise of temperature and the radiation losses, and the whole thing panned out that from the distillation of one ton of tar—he was speaking of horizontal tar—there were about 350,000 B.Th.U. available for pre-heating. He mentioned this to emphasise the extreme importance of this type of still. There was one other point which they nearly got in the discussion but not quite. He would like to know whether they had considered sufficiently the loss of heat, in distilling tar, which went up the chimney. Everyone who ran steam boilers was told to put in CO_2 recorders and so on, in order to be able to reduce the loss up the chimney. He had made some experiments on tar-stills and was horrified to find that 60% of the available heat of the fuel went up the chimney, and only 40% was utilised in distilling the tar. That, of course, was in the seating and was due to the fact that the still had to be humoured to get the real distillation. He mentioned this because if the work that Mr. Walmsley was doing on continuous distillation came to fruition they were going to have some very big savings in fuel owing to their being able to control the chimney gases very much more efficiently than was the case in a still which had to be humoured in order to get the distillate in the proper form. The one great point, as he had already mentioned, was the arrangement of the plant and the single stream right through. That was a very great point in all chemical engineering and had been lost sight of more in tar works than anywhere else.

Mr. Walmsley, replying to the discussion, said in answer to Mr. Nicholls, that both horizontal and vertical retort tars were dealt with together. He would not have much horizontal retort tar in a very short time as three of the gas works would be on verticals and only one on horizontals. Therefore, it had not been thought worth while separating the tar. He did not know that he could answer Mr. McLauren's questions, because he (Mr. McLauren) knew so much more about low temperature carbonisation than he did himself. Mr. Parrish had talked about time contact and suggested the spiral arrangement of flues. He had only had one experience of that, but the still was cut to pieces in a very short time, and he had been off the spiral arrangement ever since. Whilst they might not get the same heat efficiency in the system he had adopted, they had to take into account the length of life and wear and tear on the still, and at present stills were expensive. He did not think they would get emulsification with the typhoon agitator. He had not used it for washing oils for tar acids, but in his use of it for benzol rectification he had not had troubles with emulsification. In washing oils for tar acids, if the caustic soda were kept warm, there was a much better separation. There was some emulsification where they had dirty oils, but that could be avoided by having a small tank holding 100 or 200 gallons from which they ran off the dirt that had emulsified, and put that back with the next batch of caustic soda in the next washing. Mr. Parrish also asked for information as to fractionating columns when results had been obtained. There were any

number of columns on the market as well as opinions as to the usefulness of them, and that was the reason why he took three different types in order to get to know which was the best. When he had completed his trials, he would be very pleased to publish the results. Mr. Potter referred to the Wilton still and to the maintenance of the second coil. He himself had dismantled the top portion of this second coil used in the experiments and found from $\frac{1}{4}''$ to $\frac{3}{8}''$ of carbon on the top lap; about $\frac{1}{4}''$ on the next three and about $\frac{1}{8}''$ on the next two laps, and this process would be no use until that could be overcome. He was easily able to get pitch at 120 through the two coils alone without the aid of a final steaming coil. He got about 27% of pitch by difference as against 44%, and the funny part about the business was that when this pitch, which was 120 twist point, was examined under a microscope, together with a sample of pitch from the ordinary distillation, he could not find any more evidence of free carbon than before.

He had brought along some samples of the tar acids distilled into the various fractions and a sample of the residual pitch left from both oils at 360° C. because he thought some tar entrainment had taken place, but it had not. The temperatures employed could not under any consideration be called cracking temperatures, but he had hoped that possibly some of these complex phenol bodies might form condensation products and give something in the nature of resins. That had not happened and the tar acids were the same as before. However, he proposed to give these tar acids a chance because one did not know what might happen because they were protected by other oils and by the pitch. Therefore, he proposed to take these tar acids after extraction and put them through the coil and see if by any possible means they could get these bodies to break down in any way or cause them to condense and give something of a resinous nature. Mr. Potter had mentioned Wilton coils for treating 30,000 gallons per day with two men per shift. On the block of tar stills which they had seen at Provan there was one man on each shift. A block would do 200 tons a day, which showed a saving, as far as man power was concerned, of three men for the day on a total of 150 tons of tar as against 200.

He was glad to have the figures as to the heat which went up the chimney and of the available heat from the vapours issuing from the towers. These were things which would have to be considered, and an endeavour made to cut them down.

The vote of thanks to the author was carried with acclamation, as was one to Mr. Coleman for presiding during the Session.

An adjournment was then made, and a further Session was held in the evening.

EVENING SESSION.

At the evening session Mr. J. Arthur Reavell, M.I.M.E., M.I.Chem.E., Chairman of the Group, presided. The first paper was

THE GENERAL PROBLEM OF EVAPORATION.

By PROF. J. W. HINCHLEY, Wh.Sc., A.R.S.M., F.I.C., M.I.Chem.E.

The common meaning of the term "Evaporation" is the vaporisation of a liquid at some temperature below its boiling point, but in the technical sense the word may be considered to be synonymous with vaporisation.

In chemical factories the process of evaporation and the inverse process of condensation is taking place continuously. The subject is a very difficult one and challenges the chemical engineer at every turn. It may be considered under two heads:—

(*a*) Evaporation below the boiling point of the liquid evaporated and

(*b*) Evaporation at the boiling point.

The study of the former has occupied the attention of meteorologists, physiologists and physicists to an extraordinary extent for many years, but unfortunately the work has rarely had any chemical engineering aspect, and its utility for this purpose is generally lost sight of.

A consideration of evaporation from the point of view of the kinetic theory—the escape of molecules from the surface of a liquid through striking that surface at a high velocity—leads to some interesting and practical points. The rate of escape of such molecules is greater when the surface of the liquid is convex than when it is plane, and still greater than if the surface were concave. Above a convex surface the vapour tension must be greater and above a concave surface it must be less than that above the plane surface of a liquid. This difference in vapour pressure may be shown to be equal in dynes per sq. cm.

$$= \frac{\sigma}{\rho - \sigma} \quad \frac{2s}{r}$$

where

σ is the density of the vapour.
ρ is the density of the liquid,
s is the surface tension of the liquid,
r is the radius of the surface.

Although the effect appears to be a small one it is of importance in many kinds of evaporating plant, for evaporation is essentially a surface question. In drying plant also the physical character of the material, its porosity, etc., have an enormous influence on the rate of drying.

Dalton, Apjohn, Maxwell and Stephan are the most celebrated of the host of workers who have studied this subject. Stephan showed that the rate of evaporation from a circular flush tank of radius a is given by the expression:—

$$4\ a\ k \log \frac{P - p_0}{P - p_1}$$

where

k is the coefficient of diffusion of the vapour,
P is the total barometric pressure,
p_0 is the partial pressure in mm. of Hg. of the vapour of the air,
p_1 is the partial pressure of water vapour at the temperature of the water surface.

If p_0 and p_1 are small in respect to P this formula becomes—

$$4\ a\ k \left(\frac{p_1 - p_0}{P}\right)$$

Such a formula is useless to the chemical engineer, since it is almost impossible of application. It states that the rate of evaporation is proportional to the linear dimensions of the tank.

A short experience with figures derived from industrial work rapidly proves that the rate of evaporation may be taken to be proportional to the area of the surface without serious error, provided that other factors such as the size, the ventilation and other points with respect to the factory are taken into consideration.

An excellent account, from the physiologist's point of view, of his own and similar work on this subject is given by Dr. Leonard Hill in "The Science of Ventilation and Open-Air Treatment," published by H.M. Stationery Office.

The urgent necessity some years ago of estimating the outputs of crystallising plant compelled the author to determine the rate of evaporation from water surfaces under ordinary factory conditions. The ordinary statement that the rate of evaporation in still air was proportional to the difference between the vapour pressure of the liquid and the vapour pressure in the air was found to be untrue, but a simple and reliable formula, at any rate for chemical engineering purposes, was found and adopted. The formula may be subject to criticism on account of its dimensions, but its utility justifies its existence. Since that date hundreds of experiments with simple apparatus have been carried out by the students of the Imperial College in the course of their training, and it is found that the rate of evaporation may be expressed by a simple formula:—

$$\text{Rate of evaporation in kilograms per sq. metre per hour from water surfaces} = \left(\frac{p_e - p_d}{50}\right)^{1 \cdot 2}$$

where

p_e = vapour pressure of the liquid in mm. Hg. at the temperature of evaporation.
p_d = vapour pressure of the water vapour in the air in the same units.

This equation has been drawn in the form of a curve (Fig. 1), and as an alignment chart (Fig. 2) by Mr. Umanski (a former student of the Imperial College). Knowing the temperature of the water and the hygrometric state of the air, the formula enables one to determine at any instant the rate of evaporation from tanks of water in still air. With salt solutions it is obvious that the formula should give accurate results provided that the reduction of the vapour pressure of the solution through the presence of the salt be taken into account. These experiments have been made both with water and with salt solutions and compared with factory results. In general the agreement is good, but for estimating purposes the formula may be assumed to be 10% high.

When calculating the value of p_e for a salt solution using Raoult's law (the lowering of vapour pressure is proportional to the mol perunitage of the dissolved salt present), any dissociation of the salt should be taken into account.

Above 60° C. the experimental results are lower than those indicated by the formula owing to the fact that p_a is not the true vapour pressure in the

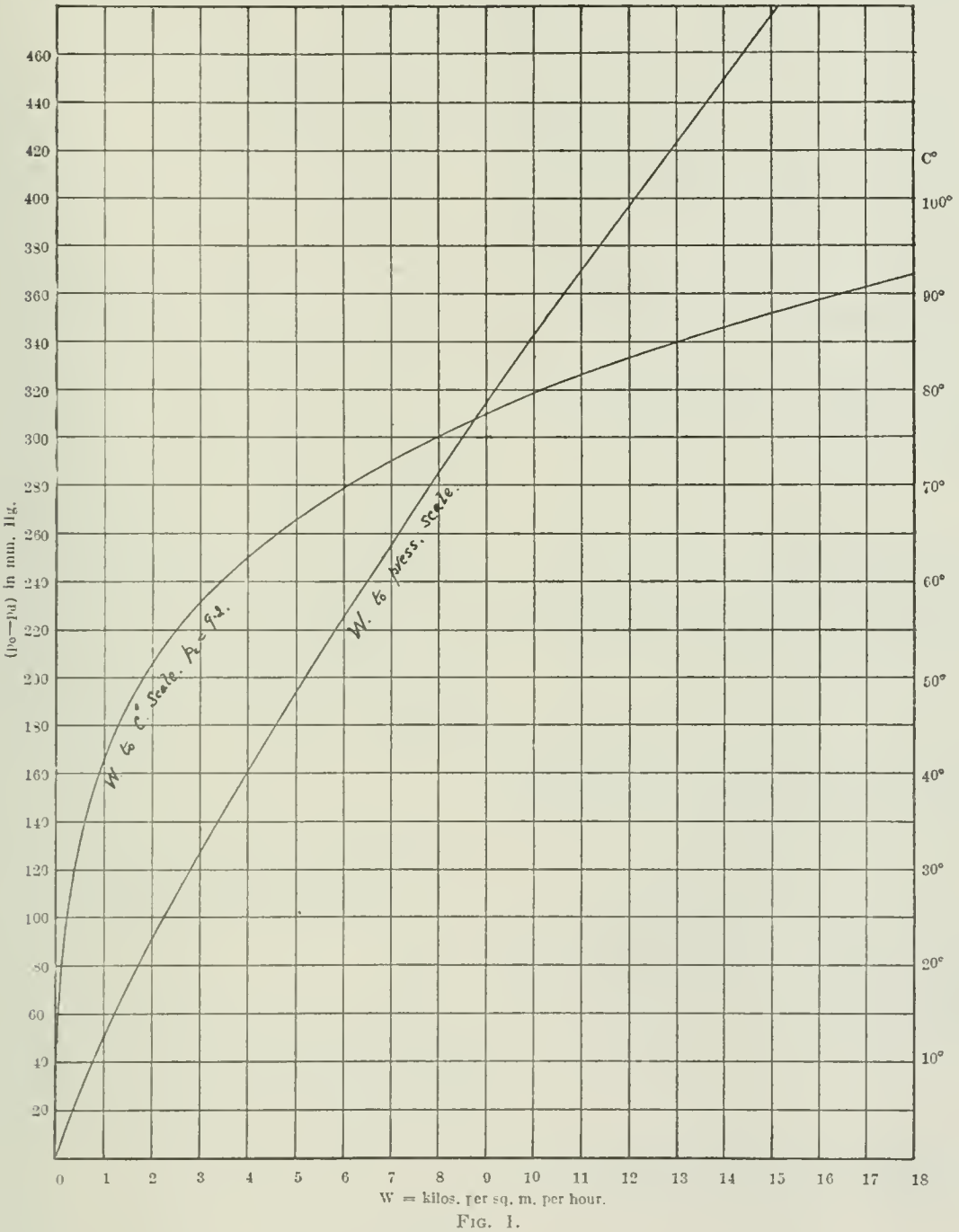


FIG. 1.

air in immediate contact with the liquid. On the other hand the margin conditions modify the effect as between large vessels and small vessels to a small extent.

It may be remarked that since from a theoretical point of view the rate of evaporation in a still atmosphere should be proportional to the vapour pressure difference, some explanation of the index is desirable. The explanation seems to be that absolutely still air is impossible under the conditions, for moist air is lighter than dry air, so that convection must be a normal accompaniment of evaporation. Again, when there is a difference of temperature between the liquid and the atmosphere the convection effect must be still more evident.

Leonard Hill, in approaching the subject from a physiological standpoint, obtained a slightly different formula. He used a simple and ingenious instrument called a "Kata-thermometer," simply an alcohol thermometer with a large bulb which measures its own rate of cooling. By covering the bulb with a "muslin glove," after dipping the whole instrument in hot water, its rate of loss of heat could be determined in any air conditions imposed. He referred all his results to the temperature of the human body. It will be obvious that the area of surface of a "muslin glove" could not be a very accurate figure for our purpose, although it is remarkably valuable and satisfactory for physiological purposes. He obtained the formula—

$$E = 0{\cdot}085 \left(p_e - p_d\right)^{\frac{4}{3}}$$

where E is the rate of evaporation loss in heat units—millicalories per sq. centimeter per second. It will be noticed that the index is $\frac{4}{3}$, not 1·2.

The work of Carrier in America calls for special mention, since he has approached the subject from a chemical engineering point of view and has obtained extremely useful results. His formula for evaporation in still air is—

$$R = 0{\cdot}093\ (e^1 - e)$$

where R is the number of pounds of water evaporated per sq. foot of surface per hour and e^1 and e are the vapour pressures of the liquid and in the air respectively in inches of mercury. Converted into metric units and using the same notation as before we get—

$$W = 0{\cdot}0178\ (p_e - p_d)$$

It will be noticed that 0·0178 is nearly $\frac{1}{56}$

Effect of velocity.—The effect of a draught on the rate of evaporation from the surfaces of liquids has been investigated by many observers. There is a difference of opinion as to whether the rate of evaporation is increased in proportion to the velocity or in proportion to the sq. root of the velocity of the current of air.

Fitzgerald in 1886 gave the formula—

$$E = \frac{(1 + \frac{1}{2}v)\ (p_e - p_d)}{60}$$

where E is the evaporation in inches per hour and v is the wind velocity in miles per hour.

Converted into metric units this would be—

$$W = \cdot 423\left(1 + \frac{v}{\cdot 894}\right)\ (p_e - p_d)$$

Jeffries in 1918 discussed the subject mathematically and found that the rate of evaporation from a circular surface of radius a =

$$2{\cdot}44\ D\ (k\ v\ a^3)^{\frac{1}{2}}$$

where D represents the fraction of the density of the air due to water vapour at the surface of evaporation.

k is a co-efficient of "eddy diffusion"

v is the velocity of the wind which is parallel to the surface.

His results appear to be in fair agreement with several observations. The conclusions he draws are:—

(1) The rate of evaporation is proportional to the sq. root of the velocity.

(2) The total evaporation from surfaces of the same shape and orientation to the wind are as the $\frac{3}{4}$ powers of the respective areas.

Several observers have adopted a formula in which the increase of evaporation due to wind is proportional to the velocity. This formula may be

$$W = (CP + Bv)\ (p_e - p_d)$$

where C and B are constants, P is the barometric pressure and v is the velocity of the wind.

The difficulties of ensuring that the current of air is parallel to the surface, that its velocity is uniform and is measured accurately, apart from the presence of disturbances and convection effects make the problem extremely difficult.

Leonard Hill's experiment first led him to a formula involving the sq. root of the velocity, but he has now added to his "still air" formula the factor $0{\cdot}102V^{0{\cdot}3}$, where V is the velocity of the wind in metres per second. This would indicate that the rate of evaporation is doubled in a wind velocity of 0·55 metre per second.

It is obvious that the rate of evaporation will be greater for a wind impinging on a surface than for a wind parallel to a surface, and it is usually found that the rate is nearly doubled when the wind strikes the surface at a right angle. This would explain the high rate given by Hill's formula, since his "Kata-thermometer" exposes a large area of vertical surface.

Carrier states that the rate of evaporation is $3\frac{1}{2}$ times as great in a wind of 4000 ft. per minute velocity as in a wind of 1000 ft. per minute velocity, and the formula he adopts indicates that the increase in evaporation is proportional to the velocity:—

$$R = \cdot 093\left(1 + \frac{v}{230}\right)\ (e' - e)$$

where

R is the rate of evaporation in lbs. of water per sq. foot per hour,

v is the velocity in ft. per minute.

e' - e are the vapour pressures of the liquid and in the air in inches of mercury.

Converted into metric units this expression becomes—

$$W = 0{\cdot}0178\left(1 + \frac{v}{1{\cdot}17}\right)\ (p_e - p_d)$$

It will be seen that in a current of air parallel to the surface the evaporation is increased to twice that in still air at a velocity of 1·17 metres per second—almost exactly half that given by Hill's formula. The effect of changes in barometric pressure on the rate of evaporation may be allowed for by modifying the formula as follows:—

$$W = 26{\cdot}2\left(\frac{p_e - p_d}{P}\right)^{1{\cdot}2}$$

and if we adopt Carrier's figure for the effect of velocity on evaporation we obtain the following formula:—

$$W = 26{\cdot}2\left(1 + \frac{v}{1{\cdot}17}\right)\ \left(\frac{p_e - p_d}{P}\right)^{1{\cdot}2}$$

where, as before, W is the number of kilos of water evaporated per sq. metre of surface per hour, v is the velocity of air in metres per second, p_e and

p_d are the vapour pressures of the liquid and in the air respectively in mm. Hg. P is the barometric pressure in mm. Hg.

This formula can be approximate only, but under ordinary practical conditions where the temperature of the liquid is less than 60° C. the error should not be more than 10%.

The experimental work at the Imperial College has not reached the stage when this formula can be thoroughly criticised.

It is obvious that the formula must fail with high velocities of air current because saturation of the air will impose a limit—in fact, the straight line law suggested cannot be strictly true.

There are many applications of the fact that the rate of evaporation from a vertical surface is practically twice that for a horizontal surface—the conditions under which direct impingement of the current of air may readily take place.

In drying cakes of wet material the rate of drying is increased greatly by placing them on edge. One of the most successful air-drying peat processes depends largely on that fact.

With respect to the evaporation of liquids other than water, the rate of evaporation appears to be proportional to the molecular weight, and for liquids which are not associated the law may be stated—

$$W = 0{\cdot}48\ M \left(\frac{p_e}{P}\right)^{1{\cdot}2} \text{ (approximately)}$$

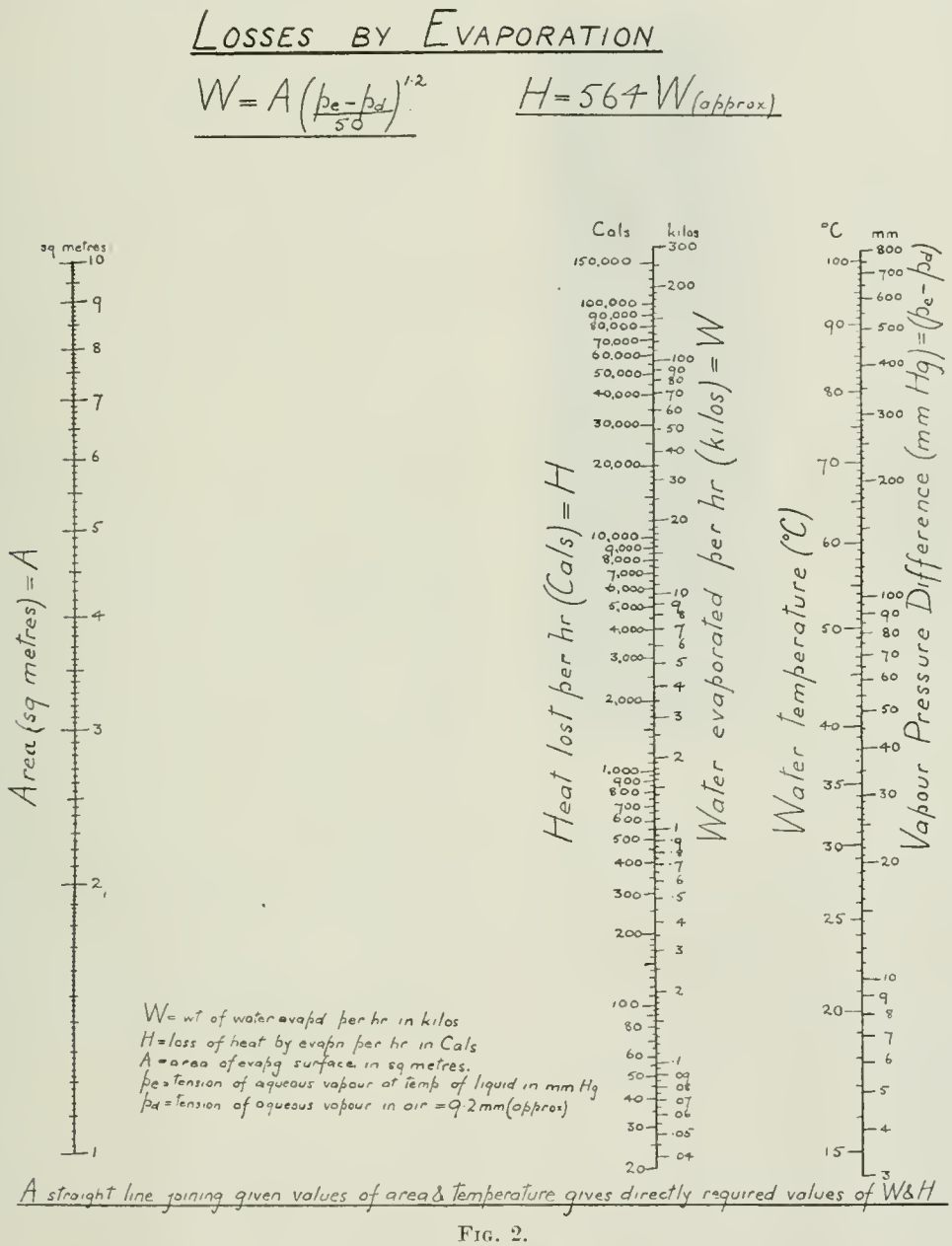

LOSSES BY EVAPORATION
W = A (pe - pd / 50)^1.2
H = 564 W (approx)
sq metres
Area (sq metres) = A
Cals
kilos
Heat lost per hr (Cals) = H
Water evaporated per hr (kilos) = W
°C
mm
Water temperature (°C)
Vapour Pressure Difference (mm Hg) = (pe - pd)
W = wt of water evapd per hr in kilos
H = loss of heat by evapn per hr in Cals
A = area of evapg surface in sq metres.
pe = tension of aqueous vapour at temp of liquid in mm Hg
pd = tension of aqueous vapour in air = 9·2 mm (approx)
A straight line joining given values of area & temperature gives directly required values of W & H

FIG. 2.

where

M is the molecular weight of the substance

p_e is the vapour pressure of the liquid

P is the barometric pressure.

W is the weight of liquid evaporated in kg. per sq. metre per hour.

Measurement of vapour pressure.—The accurate determination of the vapour pressure in the air is

becoming more and more important as the effect of air-conditioning on the output of factories is becoming appreciated. The determination of the dew point directly is often in error through the temperature or heat gradient in the instrument being comparatively great. Fig. 3 shows a convenient dew point hygrometer designed by the author. The thin silver chamber is charged with ether and air drawn through it by means of a water pump or rubber bulb. The cooling produced by the evaporation of the ether lowers the temperature of the surface until dew appears. The flow of the pump is then reduced until the dew just disappears. The mean of the temperatures at which these phenomena occur is taken to be the dew point. Unfortunately, even if the experiment is performed very slowly, and although a reading of less than one-fifth of a degree can be obtained, the temperature gradient may produce an error of twice or three times that amount. This fact is due to the high surface resistance of even such a conductor as silver, so that the temperature of the thermometer for both readings may be as much as half a degree different from that of the surface upon which the dew is formed. By making the bulb of the thermometer the container for the cooling agent as well as the surface for the deposition of dew the effect of the temperature gradient may be eliminated and a very accurate result obtained. Fig. 4 illustrates such an instrument designed by the author. Protection from the breathing of the operator may be given by a lamp-glass.

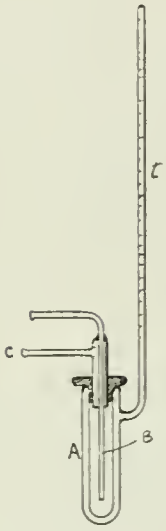


FIG. 4.

FIG. 3.

The dry and wet bulb thermometer appears to be the universal instrument of the factory for the determination of the hygrometic state of the air. A discussion of the instrument would be too long for this paper, but it may be stated that although it is nearly always inaccurate it is rarely misleading. In instruments where the bulbs are shielded and the air is drawn past the wet bulb by mechanical means most satisfactory results are obtained, but the ordinary stationary instrument is very unreliable.

The more recent electrical methods give excellent results, but if used without a complete knowledge of the principles involved may be very misleading.

Drying.—While the evaporation formula given are devised for use in cooling, crystallising, and concentrating operations, they may also be applied to drying operations in air dryers.

If the rate of drying does not exceed the rate of liquid diffusion through the material the rate of drying is nearly proportional to the perunitage of "free" moisture present in the material. The texture and plasticity of the material affect this rate to an extent which cannot generally be estimated, and experimental tests for the determination of drying factors (the relation of the rate of drying of the material to the rate of drying of a water surface under the same conditions) are always necessary.

Knowing the maximum and minimum drying factors for the material in question and therefore the maximum and minimum rates of drying the logarithmic mean of these rates will give the average rate of drying. Since the determination of a logarithmic mean is one of the commonest calculations the chemical engineer must make, I have ventured to submit a graphical method of determining it. If the smaller number be divided by the larger number and the fraction obtained be called "j," the logarithmic mean will be given by multiplying the larger number by the fraction:—

$$(1-j) \div \log_e 1/j$$

The values of this fraction are given by the curve (Fig. 5).

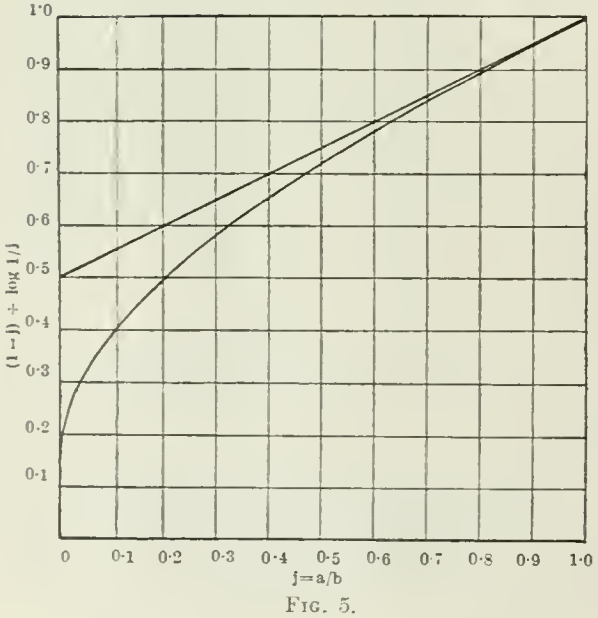


FIG. 5.

There is another point to which it is necessary to draw attention. There is a loss of heat from hot liquid surfaces which is not accounted for by the vapour evaporated.

Dr. Hill has produced experimental evidence justifying him in assuming that the loss of heat from his "Kata-thermometer" by radiation was about half the total loss.

Péclet in his classical work gives a factor for the radiation loss from water surfaces which is more than twice that from the surface of ordinary cast iron at the same temperature.

There is little doubt that these statements are over-estimates, for when this source of loss is neglected estimates on factory plant are not very seriously in error.

It is obvious that with evaporation at ordinary temperatures from the surface of crystallising vats this loss will be nil; where, however, the temperature of the liquid and air are different heat interchange takes place and the heat necessary for the evaporation of the liquid is derived from either or both according to the conditions. Generally, the temperature of the liquid tends to fall to that temperature just above the dew point at which there is equilibrium between evaporation, conduction, convection and radiation.

Boiling point evaporation.—In considering the subject of evaporation at the boiling point we meet with many new conditions and limitations to the rate of evaporation possible or desirable. A successful evaporating plant is one in which for a given quantity of heat supply the maximum rate of evaporation is produced at the minimum cost. On this account it is necessary to employ heating surfaces at their highest rates of working, to use sizes of vessels and pipe work so that for a given expenditure in capital, fuel and labour, the maximum amount of profit is obtained.

There is no question that the efficiency of chemical plant is capable of enormous improvement by the application of sound chemical engineering science both in design and operation. Few are aware to what a considerable extent this improvement may be carried.

An increase in the supply of sceptical and well-trained chemical engineers offers reward to the plant manufacturer and to the plant user as well as increase in national well-being.

The guaranteed outputs of plant manufacturers are sometimes not one half of the outputs possible under skilled management. In fact, the makers know this, but realising how much they are dependent on the factories' superintendence, they dare not guarantee more than a good performance.

The rate of heat transmission in evaporating plant from steam to water solutions may vary in the same kind of plant from 300 to over 3000 calories per sq. metre of surface per hour per 1° C. temperature difference. This simple fact illustrates the possibilities of sound chemical engineering.

In plant in which evaporation takes place from the surface it is obvious that the area of that surface should bear some relation to the amount of evaporation. The equation we have already discussed fails when $p_e = P$ (*i.e.*, at the boiling point), but it suggests that at the boiling point of water the rate of evaporation in still air would be about 26 kg. per sq. metre per hour and that such an evaporation would be produced from water without any disturbance of its surface, that is, without any boiling. This figure seems to indicate the rate of evaporation from any water solution at which no difficulties from frothing, entrainment, or other troubles could occur.

The maximum rate at which evaporation might take place from an evaporator is not easy to determine, but everyone who has had charge of evaporators or stills will have experienced the effect of projection of the liquid from the still, either as a whole or by frothing or by ordinary entrainment. The designer of the plant, however, must fix a limit to the rate of evaporation from the surface in his particular apparatus, and it has been the author's practice to suggest the figure of 250 kg. per sq. metre per hour. In rectifying plant, where entrainment must be avoided at all costs, this rate should not exceed 100 kg. per sq. metre per hour. These figures do not represent the maximum rate at which any apparatus may be worked, but they do give a convenient design figure.

The average rate of evaporation that was obtained from a surface combustion boiler designed by Michael Longridge at the instance of Professor Bone and tested at Skinningrove Iron Works was 600 kilos. per sq. metre per hour. In ordinary evaporating plants projection or serious entrainment may occur at rates exceeding 500 kilos. per sq. metre per hour.

Mr. H. J. Pooley, of Messrs. Geo. Scott and Sons, Ltd., has kindly allowed me to take figures from his own practice, and I find that in water evaporators the rate of evaporation is approximately 215 kilos. per sq. metre per hour. With glue liquors the rate works out to about 95 kilos. per sq. metre per hour; with caustic soda liquors the rate works out at 78 kilos. per sq. metre per hour and with malt extract 43 kilos. per sq. metre per hour.

In the practise of Messrs. Fullerton, Hodgart and Barclay, Ltd., however, for glycerine and caustic soda and similar liquors, the area of the pan corresponds normally to an evaporation of 150 lbs. per sq. foot per hour, or about 720 kilos. per sq. metre per hour. In this case a very high vapour space is provided to prevent entrainment.

In that type of evaporator in which the liquid is forced through tubes and the contents of the tube are projected into the chamber in which separation of liquid and vapour may take place, this limitation of area does not arise. In this type we obtain a more or less fine spray in which the convex surfaces of the drop present a maximum area and the best conditions for vaporisation On the other hand, however, frothing of the liquor may impose a working limit.

An interesting feature about frothing is the fact that there is generally a critical rate above and below which the plant may be worked without any trouble from this source.

An effective "save-all" or arrangement for the separation of spray from vapour makes the question of area of pan of less importance, but it is always desirable to measure the amount of entrainment, which is occasionally excessive.

In most commercial plants it is obvious that the limitation of the rate of evaporation is mainly a question of heat transmission. On this subject it is quite easy to produce observations which are mutually contradictory, unless one takes into account secondary effects which are neglected and generally misunderstood.

It is generally assumed that the rate of heat transmission through any given surface is proportional to the temperature drop, and this assumption enables the chemical engineer to make calculations for design purposes which are usually satisfactory in practice.

It has been stated by many observers that the rate of heat transmission per degree Centigrade of temperature difference per unit of surface increases with the temperature drop. On the other hand, there are quite a number of observations which would suggest that the rate of heat transmission is greater with small temperature drops.

In many experimental results the figures obtained are simply measures of the rate of supply of heating agent to the apparatus, the capacity of the pipe-work to supply steam, etc.

Contradictory results may be explained by simple physical facts that have been overlooked, errors in determination of mean temperature and temperature differences etc.

A few preliminary experiments at the Imperial College gave curves of heat transmission which could only be expressed by a cubic equation. It is obvious that contradictory conclusions could easily be drawn from such experimental results if

they had not been carried far enough to reveal their character.

Heat may be supplied for evaporation in many ways. With hot air in spray, tank or other simple evaporators, we have an easy proposition, and we can readily realise that the temperature of the liquid can never be very different from the dew point of the hot air in contact with it. In fact, the evaporation is really conducted at a low temperature as far as the liquor is concerned, although the temperature of the heating air may be very high. In this case, unless a dry powder is to be produced, there is much less risk of overheating than would be supposed.

Other methods of heating consist of using a direct fire or flame, hot fluids such as oil, steam and, of course, electrical methods.

A slight consideration of heating by steam is all that is possible in this paper.

It is mainly found that the amount of heating surfaces is too great for a given purpose, and wise manufacturers prefer to ensure such ample heat transmission and leave the regulation to the user.

With worm heaters excess of heating surface is more common than with the tubular heaters, but in both cases, unless the plant is in skilled hands, trouble may ensue by excessive entrainment, frothing or etc.

The simple device of fixing a diaphragm (at the flange of the steam valve, preferably) enables one to fix the maximum consumption of steam to a reasonable limit, and this device is used by many to-day, in order that plants may be left in unskilled hands without risk. It is a well-known fact that the flow of steam through a circular orifice is a maximum when the ratio of the pressures on each side of it is about ·58. This maximum flow of steam may be readily determined, and a modification of Napier's formula may be suggested for finding the approximate diameter of the orifice for a given supply of steam:—

$$d = \cdot 157\sqrt{\frac{W}{P}}$$

where

d is the diameter of the orifice in inches

P is the absolute steam pressure in lbs. per sq. inch.

W is the weight of steam in lbs. per hour.

After testing, the orifice is enlarged until the desired regulation is obtained.

Now the rate of heat transmission from steam to boiling liquids is determined mainly by—

(a) temperature drop and temperature level.

(b) the velocity of the liquid and of the heating steam.

(c) the density and viscosity of the liquid.

(d) the resistance of the surfaces and scale.

(e) the resistivity of the material of which the heating surface is made.

The work of Badger and others in American Universities is very valuable on these points.

Temperature drop.—There is little doubt that the rate of heat transmission is roughly proportional to the temperature drop; but since the amount of mechanical disturbance bears some relation to temperature drop and temperature level, and is limited by the construction of the plant, no mathematical relation is possible except for a particular design. It is found that with a particular type of plant or at a particular temperature level, a certain temperature drop will give a maximum rate of heat transmission. At high temperature levels this drop will be greater than at low temperature levels and with high hydrostatic heads greater than with low hydrostatic heads. With film evaporation this drop is smallest.

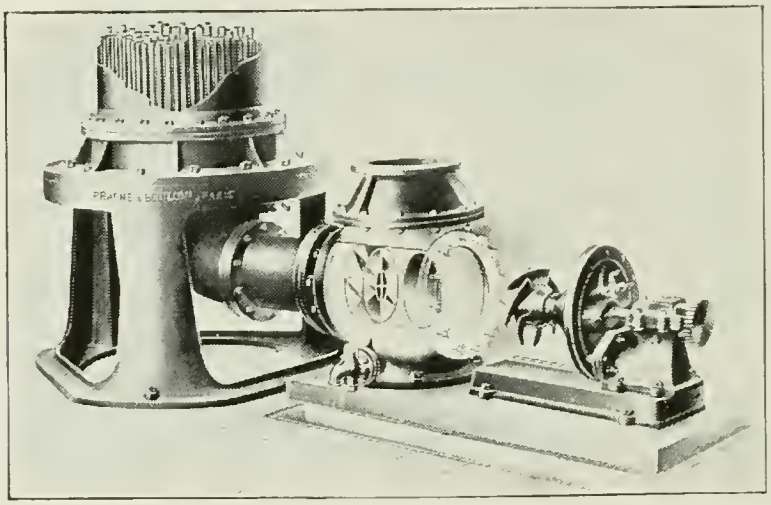


Fig. 6.

Velocity effect.—The effect of temperature drop and temperature level is bound up with (b) the effect of the velocity of the liquid and of the heating steam.

The effect of velocity on heat transmission is well known, but exact scientific knowledge is not so common.

All manufacturers and users know the value of "circulation" and the problem is approached in several ways. In the Scott evaporator a good circulation is obtained by generous design of down-comer tubes, so that resistance to flow is reduced. At the same time by keeping the heating tubes full of liquid, scaling is avoided as much as possible.

The Mirrlees Watson Co., Ltd., adopt similar methods.

In the Kestner evaporator, the generated vapour acts on the liquid like the compressed air in an

air-lift pump and enormous velocities are obtained over the heating surfaces so that a heat transmission of about 3000 calories per sq. metre per hour per 1° C. temperature difference is obtained.

The firm of Prache and Bouillon have adopted a mechanical system, and by means of a turbine pump (Fig. 6) in the base of the evaporator a velocity of about 2¾ metres per second is produced over the heating surfaces and a coefficient of heat transmission of over 3000 obtained.

Scales.—The effect of density and viscosity of the liquid on the heat transmission will be obvious without discussion, but much experimental work is needed on this point. The resistance of the metallic surfaces and of any scale formed is perhaps more important to the user than any other point. To keep clean the heat transmitting surfaces of a plant is not a question for the chemical engineer only, but for the chemist as well. The chemist can often prevent the formation of a hard scale by slight modifications of the process. The chemical engineer in charge is often powerless, and can only stop for cleaning at definite intervals—with most plants the best solution of the problem is only obtained by the assistance of the chemist.

Where the circulation is high and the heating surfaces are well submerged scale to a minimum is produced. It may be mentioned that the presence of fine solids in a well circulating evaporator will often keep the surfaces clean.

Material.—Although the effect of the heat resistivity and thickness of the material of the heating surface is much smaller than the other factors, it must not be neglected. Rates of heat transmission of over 3000 are not possible except with good heat conductors.

Multiple evaporation.—It will be realised that owing to the high latent heat of water the cost of evaporating water is very high. By using the evaporated steam as the heating agent in another apparatus a large portion of this latent heat may be recovered and a further evaporation obtained, and so on. This is the well-known system of multiple effect evaporation.

The heat pump.—Early in the last century, it was suggested that by compressing the evaporated steam, its temperature could be raised and it could be used in the same evaporator as heating steam. Siemens tried this plan in 1868, but on account of the low efficiency of compressors of his day the experiment was a failure. To-day four or five firms are making plant on this principle.

On account of the immense improvement in efficiency of electrically-driven turbo-compressors, and also by the use of high pressure steam injector compressors, a considerable amount of success has been obtained.

It will be realised that in this form of evaporation the condenser is the heating surface itself and is at a higher temperature than the liquid in the evaporator. Of course, the condensed water from the heating surface is used in a heat-exchanger for heating the in-coming liquid.

Provided that the temperature rise is small, about 5° C., the efficiency of the "heat pump" is sufficiently good. By the turbo-compressor, about 10% heat efficiency is obtained and by the injector probably over twice this amount.

It is obvious that on account of the small temperature difference (3° C. or 4° C.) the method cannot be used efficiently for concentrated salt solutions and that in any case special methods must be used for obtaining a high coefficient of heat transmission. In the Söderlund and Boberg evaporator the liquid is allowed to flow in a thin film down vertical tubes. In the Prache and Bouillon evaporator, mechanical means are adopted to move the liquid at a high speed and a very high coefficient is obtained.

Discussion.

Mr. P. Parrish said he would like to know how recent the term "heat resistivity" was, because in his days they always looked at this question from the point of view of conductivity, and not resistivity, but he could quite appreciate that the term "heat resistivity" was undoubtedly the correct one. He was very much struck with the reference that was made in the paper to the application of heat so far as evaporation was concerned. Many of them had been face to face with the problem of the concentration or evaporation of phosphoric acid. They had used lead pans with lead coils, and there had been a number of accompanying difficulties in connection with a piece of plant of that character. Not only was there the possibility of the formation of meta phosphates, but invariably there was deposition of scale, which, in a few days, reduced the efficiency of the steam coil to such an extent as to render it a very inefficient piece of plant altogether. In the heating of phosphoric acid, and then the separating of it, the use of hot air in suitable piece of apparatus got over that difficulty altogether, and, as Professor Hinchley had pointed out, it was possible to use air at a fairly high temperature and yet get no difficulty from the point of view of decomposition. There was one other matter that he desired to draw attention to, and that was whether de-frothing agents had ever been used in connection with these plants, whether frothing of the liquor imposed a limit so far as the working capacity of the plant was concerned. He raised that question because—although it was not quite a parallel case—in connection with the production of hydrochloric acid it was found that the rate at which reaction took place at the outset was such that there was entrainment, and they got a frothing over. It had been the practice for fifty or sixty years to use half a pound of tallow in connection with a charge of 10 cwts. of salt, when frothing disappeared completely. It seemed to him that there should be some suitable de-frothing agent capable of employment which would get over some of the difficulties to which reference had been made.

Mr. H. J. Pooley said the author had stated a certain figure for the rate of evaporation in plants working at the boiling point. The figure of 250 kilos per square metre was given, but that was qualified by the statement that this figure could not be applied in all cases. He did not think the author made that qualification strong enough. Very much depended on the nature of the liquor: the surface tension, the viscosity and the specific gravity might make that figure vary enormously. He himself had had under observation a plant which was giving as much as 2700 kilos of evaporation per square metre, and the fact that that took place without entrainment was proved to them quite clearly by the demonstration figures that were called for, that the distillate—it was a distilled water plant—should have a purity in solids of not greater than 0·01 per cent., and that that guarantee was carried out. It proved, therefore, that with a liquor which had twice the surface tension, such as water, the rate of evaporation might be very great indeed. On the other hand they knew that in other cases, as, for instance, glue liquor or milk, with a low surface tension, that figure might be very greatly reduced. Then again, in connection with frothing, the question of velocity should be taken into account; the velocity with which these particles were ejected into the vapour through space, the impingement of these particles one upon the other, might break the

surface or film, and in that way not only increase the evaporation, but help to avoid the loss, because they were not then carried over by the velocity of the escaping vapours.

Speaking with regard to the heat pump, Mr. Pooley said it would be very interesting if Professor Hinchley could give some further information in regard to this, as it was rather a commercial problem. Professor Hinchley had touched upon the point of temperature drop, and stated that it might, for convenience, at any rate, be said that the transmission of heat was in direct ratio to the temperature drop. It was agreed, he believed, that the heat pump was a totally impracticable proposition if the temperature conditions were increased beyond 5 to 8 degrees. He believed 5 degrees had been taken as a maximum, and that being the case, and taking the average conditions under which one might evaporate or exhaust steam to practically absolute pressure, between limits, there was a temperature difference of at least 60 degrees. Carrying that out to its logical conclusion, it would mean that one would have to have 12 effects in series; in other words, the heat pump would have to be 12 times the size of any one of the effects. That had to be borne in mind very strongly when selecting an evaporator of that type. There were certain circumstances, such as where one had a free power supply, where the purely mechanical heat pump might have very strong advantages, because there the operation would cost practically nothing, and the fuel figure would probably outbalance every other figure.

Mr. A. E. Malpas said it had struck him, from Professor Hinchley's remarks, that the crystallisation curves were drawn without reference to radiation from the side of the tank, although he confessed he was not yet clear about the formulæ. In crystallising they had two processes going on simultaneously. There was loss of heat in the mass of the liquor by evaporation from the surface of the liquor, and there was also a relatively large loss by conduction through the sides of the tank. Perhaps Professor Hinchley, in his reply, would say a little more on that point.

Mr. A. E. Alliott said, with regard to the question of surface evaporation, he would give an illustration of some of the factors which were involved from a practical example. Taking a weak liquor which tended to foam very much when it was in a very dilute state, such a liquor as sodium tungstate and carbonate of soda combined in a very weak solution, it was sometimes found that it tended to boil off even when the boiling was very gentle. A certain amount of bubbles formed on the surface, and these bubbles accumulated, and one had to go very steadily until one had got a certain amount of concentration, when that trouble disappeared and did not occur again. In carrying out such an operation he had had rather an interesting experience. He was dealing with a weak liquor, with ordinary exhaust steam and quite a low vacuum, such as 12 to 15 inches. He found that he could not concentrate at all, but, taking steam at a much lower pressure and increasing the vacuum to 28 inches, he got a thorough circulation and a big breaking up of the surface of the liquor, and the steam coming away, with, he supposed, a much more explosive effect. At any rate, much more disturbance of the liquor surface was caused, and even with that same liquor, which, under the other conditions that he had mentioned, foamed very badly, he could evaporate quite rapidly and get a sufficient amount of concentrated liquor to start the plant going in the ordinary way. Mr. Parrish had enquired whether there was any medium by which foaming could be prevented. He did not know that there was anything he would like to recommend anyone to use in the ordinary form of evaporator, because things that might prevent foaming had some drawbacks in other directions. In Canada, where they evaporated maple sugar solutions in open shallow pans, they had this little dodge. They tied a stick on to a string, and, on the point of the stick, they placed a piece of mutton or bacon fat, and brought this to a point above the centre of the pan. They then put the fire under the pan and left it. In due course the liquor started to boil, and the bit of fat melted and formed a film across the liquor, and foaming promptly disappeared. He had heard of tallow being used to stop foaming in an evaporator, but he would not be happy in recommending it. With regard to transmission through tubes, he thought it was quite certain that as the heat drop across the tube increased they did not of necessity get a proportional evaporation. There were so many factors to be taken into account that it was difficult to make an *ex cathedra* statement to cover everything, but it seemed that one could get a comparatively low temperature drop which would give almost as rapid an evaporation as was obtainable with a very high one. That low temperature drop, however, was not of the order of 5° C., as was suggested in the paper for the heat pump. One factor which ought to be mentioned was the use of very long tubes full of liquor, when they had got a high vacuum on one side and the steam itself was well below atmospheric pressure. Then, of course, they had to take into account the fact that the vacuum was affected by the head of the liquor in the tubes, and unless they were careful to watch that the lower portion of the tubes might not be able to give any boiling effect at all; in fact, it was reduced to a mere transmission of heat from steam to liquor which was not at its boiling point, and was very ineffective; in fact, it might be said that the bottom portion of such tubes was doing no work at all. Then one came to the question of air elimination, which was a very important factor. If air remained in the heating surface on the steam side they were not getting the temperature which was due to the pressure shown, and the efficiency of the evaporator might be cut down in consequence. He thoroughly agreed with Professor Hinchley when he said that the heat transmission through the material of the tubes themselves was perhaps the least of the resistances which the heat had to overcome. The surface effect was big, and, of course, they were always up against the fact that if they had any fat or grease or oily substance in the liquor which was being evaporated, or if there were in the solution any substance capable of forming a film and which tended to lower the surface tension, then this substance, both by the ordinary law of surface energy and also by the law of the general contrariness of inanimate substances, would act on the heating substance and cause scaling to occur.

Dr. L. F. Goodwin said he had been engaged as a chemist for two years in a very large Canadian paper pulp works, studying the problem of evaporation and general efficiency. He had found that the efficiency of many of the quadruple effect evaporators was cut down enormously by frothing. There were cases, in California, of potash evaporation which took 14 hours to work off a certain quantity, but by eliminating frothing, by means of a defrothing agent, that time had been reduced by 4 hours, and a good deal could be done in that way. It was not much good having an evaporator which would do three or four times what it was actually doing by eliminating frothing. Referring to the heat pump, he had found that, in spite of extraordinarily cheap electrical power, it was not going to pay to use this apparatus, although he had heard of three or four plants which were working successfully. He hoped Professor's Hinchley's experiments would give some accurate figures on the point.

The following letter was received at the meeting, and the Chairman stated it would appear in the printed record:—

"Referring to the heat pump, in the 'P and B' Evaporator, the form of injector compressor used is such that a temperature rise of 18° C. can be obtained if the design makes it necessary, and at this figure the efficiency of the thermo-compressor, as it is called, has still a very considerable advantage over any mechanical type of compressor. With this temperature rise it is possible with suitable design to instal as many as nine effects, each working with a 2° C. temperature drop, and with the ninth effect, of course, working under atmospheric pressure. In an experimental apparatus so constructed a total evaporation of 12 lb. of gained water per lb. of live steam used has been obtained.

"In an ordinary single effect plant where the tubular element is of necessity both condenser and heating surface, the firm handling the 'P and B' Evaporator are always prepared in the case of water distillation to guarantee that 4¼ lb. of water will be evaporated per lb. of live steam used. This result is comparable with the output of any quadruple effect evaporator working from the atmospheric pressure downwards, and the gain in first cost and space occupied is evident.

"It is not possible to define a successful evaporator without some reference to its non-scaling properties, and unless a plant can be manufactured and operated so that scale is positively prevented from forming on the heating surface no output figures based on tests carried out in the clean state are of much interest to the user.

"The writer's experience is that the proper solution of this difficult question is to be found by adopting rapid turbulent circulation and controlled ebullition with, in a few cases, the addition to the circulating liquid of an accurately measured quantity of chemically innocuous abrasive matter.

"In the 'P and B' plant speeds of circulation even greater than those mentioned by Professor Hinchley are obtained by means of the special construction illustrated in the paper, and while a co-efficient of heat transmission of 3000 is used for design purposes, figures considerably in excess of this have been observed under test conditions."

Professor Hinchley, replying to the discussion, stated he had adopted "heat resistivity" instead of "heat conductivity" as a basis of discussion because one added resistivities, and the calculation became simple. On the question of entrainment, 3 per cent. was quite common, and 1 per cent. was a regular thing.

He congratulated Mr. Pooley on the result he had obtained. He was quite sure that Mr. Malpas's method of putting down a few plants and finding out what happened was a method which the consulting chemical engineer dare not adopt. He had to state exactly how much work the plant would do before it was made, and what the formulæ in the paper provided was a means of knowing what was going to happen without carrying out these experiments. The curves and the formulæ in the paper were an attempt to enable them to find out the number of crystallising tanks required for a given job.

With regard to air, he might mention that air was heavier than steam at the same temperature, and that the proper place to let the air out was generally near the bottom and not near the top of a vessel.

The whole object of the paper was to instil accuracy, consistent with cost.

On the motion of the Chairman, a hearty vote of thanks was accorded the author for his paper.

The final paper of the Conference was:—

HISTORICAL DEVELOPMENT OF DISTILLATION OF GLYCERIN.

By T. H. GRAY.

The object of this paper is to present a short account of the development of the manufacture of glycerin in general, with an account of Scott's glycerin plant in rather more detail.

Turning to the early history of the subject we find Scheele—1779—mentions the "sweet principle of fats" obtained by him by digesting lead oxide with olive oil, the liquid product being glycerin in a dilute impure form.

Chevreul by his classic researches on oils and fats, in the period between 1813—1823, systematised the chemistry of the subject considerably. About this time glycerin was prepared solely by Scheele's method, incidentally being a substance only of scientific interest, having little or no practical value.

There were various methods given for preparation of glycerin, but more often they relied on alcoholic extraction of the liquid obtained by Scheele's process, soap lyes and fat-splitting not as yet being known. Curiously enough, we may note in passing that at this early stage charcoal was often used for decolorising the weak glycerin thus produced.

Cap's process was the first variation used for glycerin production about 1830-1840. He used the liquors from the lime saponification of tallow in candle manufacture. After appropriate treatment, the neutralised liquor was carefully evaporated and then passed through bone charcoal, when a more or less pure glycerin resulted.

Tilghmann, about 1854, passed an emulsion of fat and steam through pipes of iron, or copper, at a temperature of 600° F. (365° C.). He obtained a glycerin liquor, which, after treatment, as in Cap's process, gave a fair product, relatively speaking.

G. F. Wilson, of Price's Candle Company, also in 1854, by his patent No. 1624, made the first notable advance in glycerin production by actually distilling candle works "sweet liquor." Fire-heated copper stills were used, of a capacity of 10-30 cwt. of crude glycerin, with air condensers. For the purest glycerin the condensers were silver-plated. There were big losses in this process owing to the high temperature used, there being a lot of steam exhaust, not condensed, containing glycerin. Nevertheless, as we shall see, this type of plant, with variations, persisted till the introduction of modern steam-heated vacuum stills.

Again in 1855, Patent No. 301, Wilson and Payne improved the process. Steam was passed through weak candle liquor, concentrated in the still to sp. gr. 1·5 by fire, until all volatile acid substances were removed. The temperature was then raised, and superheated steam freely admitted (thus excluding air as far as possible), when glycerin distilled above about 340° F. (170° C.), the best results being obtained at about 370° F. (190° C.). They claimed a high degree of purity for their distillate, especially after treatment with charcoal, and no doubt they were justified. About this time glycerin began to be of some interest and importance in the arts and manufactures, and consequently became much more sought after.

The patent of E. T. Hughes calls for little comment, being No. 1459 of 1859, except that he specified the use of a lead-lined vessel as still, and condensed the vapours in a refrigerator.

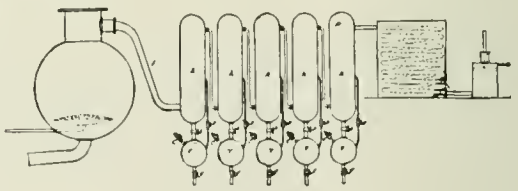
The next step of importance was so late as 1881. F. J. O'Farrell patented the use of vacuum and pump for distillation, together with steam at 200° C., using soap lye crude in the still. This lye was no doubt substantially soap crude as we know it to-day, except for purity. Most likely it would be open pan, fire-heat concentrated, and fairly impure. Nevertheless, this process marks a distinct advance, for in addition to using a vacuum, he provided two sets of receivers connected by a vacuum break cock, thus allowing of continuous distillation.

We now enter a period of some activity from the point of view of design of apparatus. The use of nitro-glycerin in blasting, railway making, and kindred objects, together with the rapidly extended use of glycerin in the arts no doubt supplying the necessary stimulus.

Following closely on the previous patent came W. Clark, of 1881, No. 5384. He also used vacuum and pump, but moreover insisted on vacuum being maintained as high as possible during distillation; claiming a glycerin of sp. gr. 1·260 against the best then obtainable of 1·160. As he used a set of column condensers in series, it is difficult to see how the patentee got quite all he claimed, as fractional condensation would occur in the series.

The next item of interest is G. Payne's patent of 1882, No. 203; who used a steam superheated condenser, temperature 150°—200° C., thus obtaining glycerin of a high gravity, the water vapour passing well forward in the condensers.

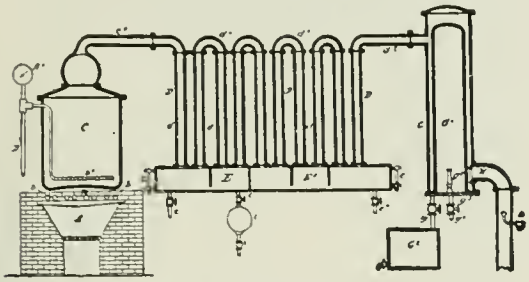
O'Farrell again came on the scene with a patent in 1883, No. 1584, which was substantially a combination of Clark's and Payne's ideas, using a similar plant with steam-heated condenser at about 100° C.—otherwise there is no point of interest to mention.



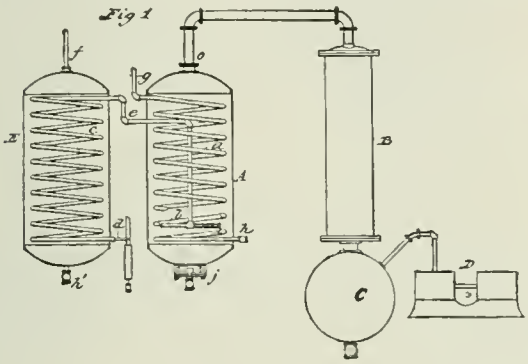
Clark's Still.

Again we meet with, in A. M. Clark's patent of 1884, No. 13,925, who used a complicated system of combined distillation and evaporation, a big plant with no particular interest except that a barometric column was included in a design for the first time. That and the small attempt to conserve steam energy are the features mainly observed.

A. G. Brookes next in 1885 (patent No. 5382) used a quite good-looking design with two pans, specifying the product in the second pan "fraction" to be fit for nitro-glycerin production. Brookes evidently used a small still, as he distilled a charge at one operation, and had the pans big enough to contain the total distillate. Another item of interest as showing the trend of thought of this time, is his specially designed condenser to hold back as much glycerin vapour as possible. At this time attention was directed to the question of efficiencies, costs and yields.



Brookes' Still.



As his original design.

Apparently the impetus of inventive effort had spent itself for a period, and a "marking time" resulted while various of the plants we have so far considered were being used.

The next step to notice is the patent of the Belgian chemical engineer Van Ruymbeke, dated 1893, No. 24,456, and two subsequent patents taken out in conjunction with W. F. Jobbins in 1894 and 1896, and numbered respectively 12,367 and 20,982. The first-named, No. 24,456, contained the master idea, and the patentee used a high-pressure steam coil for still heating, doing away with direct fire altogether. This, together with the use of expanded reheated steam for the actual distillation, marked a really notable advance in design. By these means the decomposition of glycerin was largely obviated, and though the free steam was at a low pressure, it was as hot as the steam in the heating coil of the still proper.

The two other patents above mentioned were only modifications in detail, and the only note of interest was contained in a specific mention of the way in which to eliminate glycols by a preliminary operation at lower temperatures.

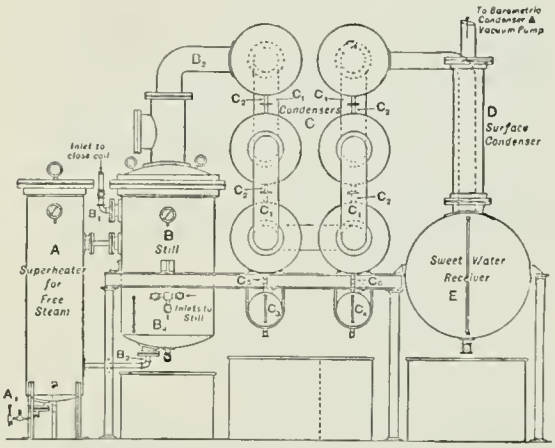
It is hardly necessary to mention that Van Ruymbeke also used a high vacuum, working pressure being about 28 in. of mercury, or even a shade higher.

As a matter of general interest I may mention the large plant at Gretna, put up by the Government during the war, was of Van Ruymbeke's design, erected I believe under his personal supervision.

Distillation figures on Van Ruymbeke plant.
Still charge = 149·26 at 82·8% Glycerol = 123·5 at 100%.

Distillate.	Cwt.	Glycerol content. %	Final yield. %
Strongs	108·5	97·5	85·7
Weaks	138·0	9·2	10·3
Foots	23·0	14·2	2·6

Total yield = 98·6. Loss = 1·4.



Von Ruymbeke's Still and Superheater.

Sectional elevation of present-day apparatus.

The next plant to be noticed is Scott's patent of 1899, No. 15,159, which combined features of both the old types and the new. His still, though set over a fire, received only a little heat therefrom, but was mainly heated by a superheated steam coil placed round the still bottom. The cooling battery was of long vertical air-cooled pipes with receivers attached, and a tubular condenser, preferably supplied with weak glycerin liquor, this becoming, therefore, somewhat concentrated during the operation of distilling. As this is a plant with which I am familiar, I propose to describe some of its working in detail.

To-day we use a modification of the original plant. A still of the usual design is set over a flue, well back from direct flame heat, which same fire heats a superheater coil for steam. This steam is led into the still by a large heating coil of pipe, eventually terminating in a length of perforated pipe, with steam outlet holes about ¼in. in diameter. By this means the crude is mainly heated, and a very small amount of thermal decomposition occurs. The crude is fed in continuously during distillation, a charge being approximately 6 tons for a first operation. The still is provided with a catch vessel of the usual type for entrainment or priming.

We are able, by means of the fractional condensers used, to obtain good strong glycerin, fit, after concentration, for dynamite, on the first four columns and receivers, whilst the last three columns give weaks—or rejects, from 2° Tw. to 14° Tw., depending on local conditions at the

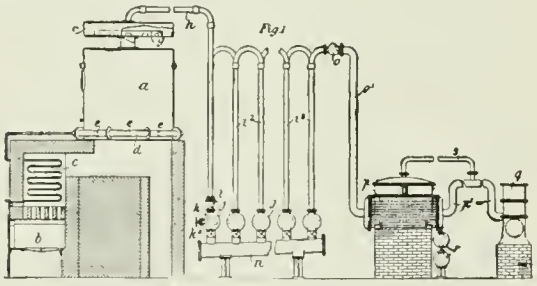
moment. These weaks are again distilled after concentration, and give a distillate fit for commercial glycerin. A final tubular water condenser ensures only steam going forward through the barometric condenser and pump. Steam from the superheater coil is adjusted by a regulator valve to 6lb. pressure, and a temperature of 250° C. vacuum being 28in. of mercury, maintained by Scott's wet type pump, the still temperature being 180° C.

Dynamite glycerin from this plant was actually the first batch nitrated at Gretna during the war, with very satisfactory results, and during the whole of the war yielded glycerin, once distilled, which satisfied Nobel's 21D Specification.

In passing it is worthy of notice that the quality of soap lye crude supplied to the still, has a marked effect upon the products and yields of distillation. For instance, we have had no troubles with trimethyleneglycol in the distillate owing to our crude being of exceptional quality for a soap lye crude.

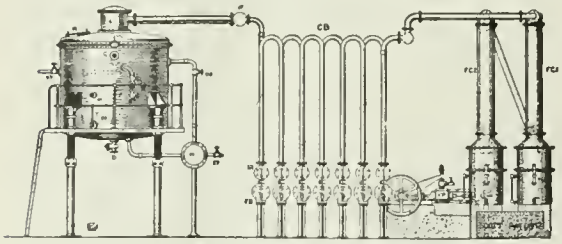
The foots left in the still after all good glycerin has passed over, are washed out, treated and concentrated to crude, and this material is separately distilled when a sufficient quantity has accumulated. This "foots crude" yields about 60 per cent. on being re-distilled as a separate charge.

Using our average crude, having glycerol I.S.M. 82·80 per cent., ash 9·85 per cent., organic residue 2·10 per cent., sp. gr. 1·3016, we obtain a yield of approximately 96 per cent. throughout a year. On small runs as high as 97 per cent. and 97.5 per cent. has been realised.



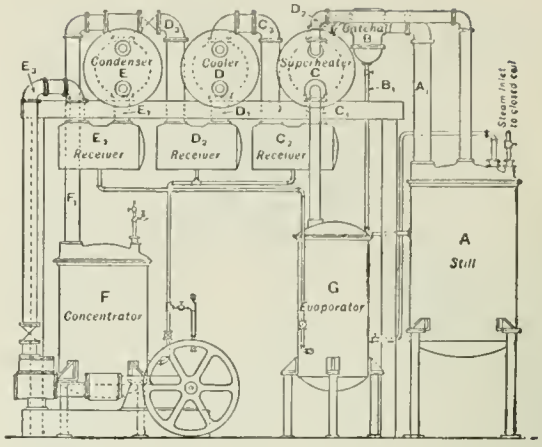
An early Scott plant.

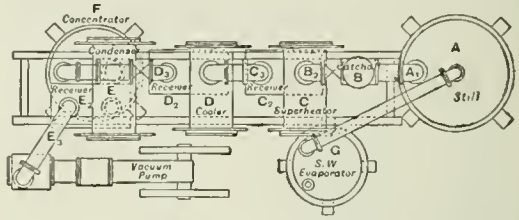
Discussing yields I would like to quote an expression of opinion given to me by one of our leading chemical engineers:—"Short period tests on any chemical plant do not carry any great conviction, however good the figures. Long period tests carry all sorts of minor handicaps, as men's carelessness, bad materials, works' mishaps, stoppages, etc., and it is very rare for them to show as good figures as those taken on test when all are on the alert, and conditions are of the best."



Scott's "New" Still.

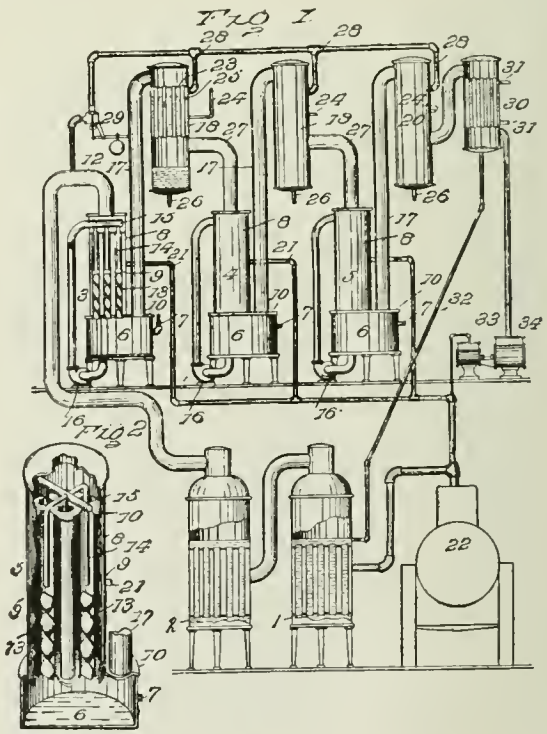
So far we have only considered once-distilled or commercial glycerin. A very important branch of glycerin distillation is the manufacture of chemically pure quality. This is usually attained by a double distillation, or, as some works prefer, even a third. This quality represents the height of





Sectional elevation of complete Garrigues plant.

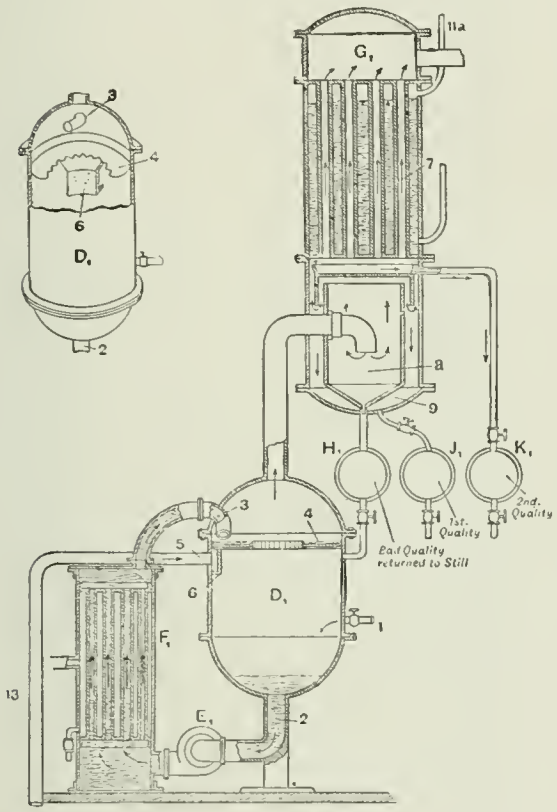
excellence in glycerin distillation, it being colourless, odourless, free from salts of metals (as iron, copper or lead), and containing as little as 0·0050 per cent. Cl. Last, but not least, there should be a very low figure for arsenic, the present official limits being four parts per million, according to



Elevation of three Wood Stills in series according to his original design, forming one self-contained unit.

The British Pharmacopœia. Incidentally it is highly probable that the arsenic present in such small ratio as we have mentioned, distils over in the form of a glyceryl ester, probably as glyceryl arsenite—simple distillation not being sufficient to eliminate arsenic completely. The further treatment of the doubly distilled material for pure glycerin is rather apart from distillation, though full of interest, particularly as regards quick removal of colour by means of charcoal of various types. As already mentioned, animal charcoal was formerly used for decolorising, and is used to-day together with various other decolorisers.

The next plant to consider is that of W. E. Garrigues of 1905, No. 24,100. This apparatus has a novel steam superheater, the hot vapours from the still passing round a combined condenser and



Detailed section through one Wood Still and Condenser.

superheater, the still, condensers, and concentrators being one self-contained unit. It is a fairly complicated design, and requires careful working to get the best results in distillation, though at the same time is economical in heat energy consumption.

Another very modern type of plant is that of F. J. Wood, in patents 1907, No. 21,658, and 1913, No. 24,920. This plant is even more intricate than any other we have considered, and the main new idea in it is to work from one to as many as six stills, with one jet of steam. The free steam in this case actually meets with a spray of glycerin from the upper portion of the still, thereby carrying forward glycerin vapours, which pass on to the condenser. This condenser is so designed as to be a tubular hot water system in the upper portion, the lower being a simple catchall. The result is, in brief, that glycerin vapour is condensed while the water vapour passes through the hot tubular part, to supply steam to another still in the series. This plant shows the greatest economy in steam consumption of any we have seen, though this can only be attained by very careful working and supervision. In this connection I would like to quote some comparative figures from the chapter on "Glycerin distillation," by Lieut.-Col. Briggs, D.S.O., in Dr. Young's latest volume, "Distillation Principles and Processes," and to acknowledge, with pleasure, much valuable criticism and figures obtained therefrom. He quotes the relative fuel efficiencies of the Van Ruymbeke, Garrigues and Wood stills respectively as 16, 14, and 13 of coal for one ton of once distilled glycerin.

As the final development in glycerin distillation we may very briefly consider the idea propounded by the "Société Française des Glycerines," in No. 125574 of 1919. This plant is so designed as to work without the use of free steam. Crude glycerin at a temperature of 180° C. is passed in an atomized state from one vessel, by means of suitable jets, to another vessel or still of higher vacuum. Steam coils maintain the temperature about 180° C., and condensers and pump are used as necessary. We have no practical details of this plant, but it is evident the idea is very ingenious, though at the same time there are some very obvious troublesome conditions involved, as for instance choking of jets and removal of the solid residues.

Distillation figures for Wood's Still.

Still charge = 100 tons crude at 80% = 80 tons.

Quality.	Amount.	Glycerol content.	Yield %
Strongs	69·5	99·0	86
Weaks	70·0	5·71	5
Foots	18·0	40·0	9

Broadly speaking, we may summarise the progress made in glycerin distillation as having gone along the following lines:—

(1). By actual improvement in design of apparatus from an engineering standpoint.

(2). Glycerin of to-day is practically a pure standard substance, in so far as a commercial material may be.

(3). To-day, as distinct from the earlier times, efficiency and economical use of energy are the points aimed at by our chemical engineers and designers.

In conclusion, my best thanks are due to Mr. Henry Simpson, General Manager of Gerard Brothers, Ltd., for his great help during the preparation of this paper.

Discussion.

Mr. H. J. Pooley said this was a very interesting account of the different apparatus, and knowing Mr. Gray as an expert in the handling of this subject, he would be glad if he would give them a little more enlightenment. He would pass over the question of fuel efficiency in plants of this kind, because in the worst of such plants the fuel cost was relatively unimportant. They were dealing with a material in which the finished product was never, under any conditions, less than £80 a ton, and the question of 2 or 3 cwt. of fuel was therefore relatively unimportant. What did matter was the question of yield and purity and rehandling, and he would like the author to say if he had any information in regard to the particular form in which one of the worst impurities in the distillate—viz., chloride, appeared, and whether sodium chloride was carried over by entrainment, or whether it was carried over by sublimation. He himself had noticed that he got chlorides down in

the earlier condensers and the arsenic in the latest or weaker condensers. The author had stated that the arsenic came over as a glycerol ester, and that would probably explain the fact of it condensing at the lower temperatures. He would also like to know if the author could give some figures as to the point at which the loss occurred; whether he had examined the foots to see the proportion of glycerin which remained as glycerin, and the amount that was destroyed actually in the distillate and which he supposed passed over as acrolein, and the amount that passed away in the final condensing water in the jet or barometric condenser. That was a point of interest to designers. He would also like to know if the author had figures with regard to the relative loss on the first and second distillations, in dealing with soapmaker's crude, which contained 10% of salt, in which case the loss in the foots was likely to be greater. He himself had noticed that in dealing with saponification crude, *i.e.*, crude from the splitting of oils other than by saponification, a distillate giving a yield of 98% was usually obtained, and that would seem to indicate that the loss on the second distillate was comparatively small. If the author could deal with these points he would be extremely obliged.

Mr. P. Parrish said the only point he wished to raise was the question of the application of oil. It seemed to him that the distillation of glycerin was essentially a distillation that ought to be done by means of the application of hot oil which was recirculated, and he wondered whether the author had considered that question. In connexion with all these complex organic bodies, where one was anxious to avoid decomposition, one reverted almost intuitively to the question of the use of hot oil and recirculation in order to obtain something approaching an efficiency. Certainly he had been struck with the antiquated nature of the apparatus and the fact that a coal fire was used in connexion with the distillation of glycerin compounds and compounds of that character under conditions where it was very difficult to control the local temperature, and it seemed to him that the use of hot oil and recirculation would reduce the complication of glyceriu distillation.

Prof. J. W. Hinchley congratulated the author on having presented a very interesting paper, but he would like to suggest that in the paper as finally printed he should expand some of the practical points. It was obvious that in the distillation of glycerin, a very high vacuum was employed. In one case, the author stated that a vacuum of 28 inches was employed. He understood that 13 or 14 mm. was quite common with superheated steam injected at the same time. The function of the superheated steam, he judged from one of the patents referred to in the paper, was obviously that of simply supplying heat to the still. The question raised by Mr. Parrish, of the use of hot oil, was a point in this connexion. He would like to know if the author had had any experience of the jet apparatus described in one part of the paper, and whether this was a common method and what was the function, because in many of these apparatus what was used for one purpose in one case was used for another in others. In this particular case, he understood it was not the wet distillation of glycerin, and that the steam was used simply to provide heat. Was that so?

Mr. Gray: Replying generally to the discussion, the author said that a short time ago there had been a disastrous fire at his works which destroyed the records of 15 years of glycerin manufacture. He had found a few sheets in clearing up the debris, and from these he might be able to get a few figures which would answer the points raised by Mr. Pooley. He agreed that the question of fuel efficiency was not of great importance, although in a general sense, perhaps, it mattered. The figures showed that well under one ton of coal was used per ton of crude. He had worked out one set of figures, and they showed that for 362 tons of crude the coal consumption was 190 tons. That, however, did not cover the whole question of fuel efficiency, because the coal did not include the steam for the concentration of some portion of the "weaks" (*i.e.*, weak liquors condensed). Generally speaking, however, they worked to a very fair degree of efficiency with fuel. The question of sodium chloride coming over he did not think he could answer. He had wondered himself what was the real reason for this. In the case of distillation from an ordinary crude containing about 10% of common salt, the usual thing on the first distillation was to find about 0·02% of sodium chloride. Although that was a very small figure, it was too much for some technical operations. He rather thought it was due to entrainment, but he would not like to say definitely, because there were some curious contradictions in the matter. The bulk of the sodium chloride was found in the first two condensers, and a very small proportion afterwards. With regard to Mr. Pooley's other point,

Mr. Gray said that on second distillation he found it was easily possible to approach 99·5% yield—*i.e.*, redistilling the worst distillate theoretically. In connexion with the final portion of Mr. Pooley's question, he had been to the trouble to collect over a long period a few tons of weak liquor from the barometric condenser, which he boiled and boiled and boiled, and the amount of glycerin produced was of no real value.

In connexion with Mr. Parrish's remarks about oil heating, Mr. Gray said he had tried to make it clear in his rough drawing that he did not depend upon a direct flame. It was merely hot air. He would, however, consider the hot oil suggestion.

In answer to Prof. Hinchley, Mr. Gray said that steam is absolutely essential to the distillation of glycerin.

A hearty vote of thanks was accorded the author at the conclusion of the discussion.

On the motion of Mr. Gee, a vote of thanks was passed to Mr. Reavell for presiding at the meeting, and the Conference closed.

THE DE-WATERING OF PEAT BY PRESSURE.

By PROF. J. W. HINCHLEY, Wh.Sc., A.R.S.M., F.I.C., M.I.Chem.E.

Peat is a material which occurs in extraordinarily large areas all over the world, and is formed by the decomposition of plants in the absence of air.

The quality of the peat is determined by the character of the plants which led to its formation, the latitude of the place and the geological formation of the country.

Since water forms the medium by which contact with air is prevented, the country where the peat accumulates is shaped like a basin, and the subsoil consists of an impermeable layer of clay or similar material.

It will be realised that there is enormous variation, both chemically and physically, in the quality of peat from different bogs, and any discussion on the treatment of peat cannot be exact in a general way, since the behaviour of the peat from different districts must vary considerably. Peaty matter contains on an average, when free from moisture, about 58% of carbon, the remainder being mainly hydrogen and oxygen, and according to the conditions under which the deposit is formed, associated ash may vary from 0·5 to 20 or 30%. It is obvious that peat will contain such chemical substances (or their decomposition products) which characterise the plants from which it is produced, and it is found that while some peats contain as much as 10% of waxes, resins, etc., others may contain less than 1% of these substances. As a general rule peat contains less sulphur than the plants from which it is derived, and it is interesting to note that this elimination of sulphur may often be observed in peat bogs. In the Doncaster Bog, for example, the light-coloured peat which is found near the surface of the bog is continually giving off sulphurous gases, while the black peat immediately under it is absolutely free from smell.

On account of its comparative freedom from sulphur, and its well-divided condition, dry peat burns with higher efficiency in furnaces than coal and does not seriously attack the metal parts of the plant. On this account peat-fired plant has a lower rate of depreciation than coal-fired apparatus. Peat carbon is a valuable raw material in industry, and it is possible to obtain from it an almost chemically pure carbon. Its finely-divided condition makes it most convenient also for the manufacture of decolorising and activated carbons. Raw peat in fairly-drained bogs usually contains from 85 to 90% water, *i.e.*, each part of the dry substance is associated with from 6 to 9 parts of water. The peat also contains a quantity of colloidal material which is useful in some processes for the utilisation of peat, whilst in other processes steps are taken to destroy it. The shrinkage of air-dried peat as cut from the bog, on account of the presence of this colloidal material, is generally about 70%.

Up to the present, the only methods of utilising peat which have persisted have depended upon air-drying. The simplest process consists of exposing sods cut from the peat bog to the air for a sufficient length of time. Variations of this process consist in " kneading " or " masticating " the peat in such a way that the plasticity is increased; so that, on drying, the contraction is greater and the density of the final product is raised.

As peat occurs in the bog, it may be taken, roughly, that one cubic metre of peat weighs 1000 kg. (say one ton), and as in the course of drying, contraction takes place, the density of the final peat may vary from 0·2 to 1. It is obvious that the quality of the peat will vary with its depth from the surface of the bog, the upper layers or youngest peat being most fibrous and the oldest peat, that at the bottom of the bog, being most decomposed. At the same time, owing to the water-logged condition of the bog, the lower layer will not only be more decomposed, but may contain colloidal material washed from the other layers. The density of the dried peat from the lower layers on account of pressure will be much greater than that from the upper layers, while the content of water is usually less. Peat from the upper layers of many bogs is of a fibrous character, and, after drying, makes a most useful " litter " for animals, and the dust gives a packing for fruit and similar goods. The products of the decomposition of the vegetable matter have disinfectant properties, which render the material of extremely great value for these purposes, whilst, after use, it is available as a manure.

The efficiency of methods of obtaining peat by air-drying is limited by the character of the climate, and in few cases can more than two " crops " per year be obtained. It is obvious, therefore, that the commercial utilisation of peat can only be small, since the rate of working from a bog is so slow. Attempts have been made and very large sums of money have been lost in machine-devices for drying peat at a sufficiently rapid rate to produce commercially an industrial fuel or other industrial products.

Now the calorific value of ashless, dried peat is, at the most, 6000 calories per kg. The best British peats have a calorific value of about 5500 calories per kg. The calorific value of peat as burnt in furnaces is somewhat lower than this, on account of the " equilibrium-moisture " associated with it, and may generally be taken at about 3500 calories per kg. Of all the processes for the rapid drying of peat, it will be clear without much consideration that a direct drying operation could not be a commercial success. In the best drying plants at least 600 calories is required for the evaporation of one kg. of water, so that the association of 1 kg. of peat with 6 kg. of water would mean that the whole of the heat energy available from the peat on combustion would be required for its drying. Recently, the problem of drying by direct heat has been resuscitated through the development of the " heat pump evaporator." Heat pump evaporators are now made which may be depended upon to evaporate at a rate such that 1 kg. of steam will evaporate 4 kg. of water. Whether this process can be applied to such a material as peat is extremely doubtful, since the difficulties associated with evaporating liquids in such apparatus are enormously increased with a material like peat.

The author has been engaged for several years past on methods of de-watering peat by pressure and is convinced that such methods offer an attractive commercial solution of the problem.

Curves showing change in pressure, thickness of cake, flow of water.

Charge 36 lb. 80·6%.
Cakes 28·5 lb. 71·1 to 79·6%.
Cold pressing.

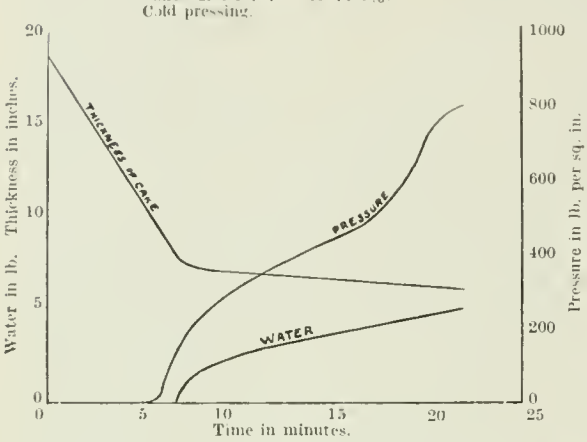


FIG. I.

In drying peat by heat or by air-drying processes, the presence of the colloidal matter may be a distinct advantage, but in getting rid of the water by pressure, the presence of this material is a most serious objection, and methods have to be adopted by which it is completely or largely destroyed. Both extreme cold and heat are capable of bringing about this result, and the problem of getting rid of the water is reduced to devising a mechanical process which shall not only be practical but will pay.

It has been stated that the percentage of water in peat as it occurs in a drained bog is usually from 85 to 90 %. By a simple pressing operation in the cold, which can be carried out by means of a "squeezing-conveyer" at the bog itself, this percentage of water may be reduced to from 80 to 81%, or four parts of water to one part of peat. A pressure of 50 lb. per sq. in. is necessary and the time of application will depend on the thickness of the layer.

Practical experiment by the author has shown that in suitable apparatus it is not necessary to destroy entirely the colloidal matter present in the peat to remove the water successfully by pressure down to approximately 50%. Ekenberg has stated that a temperature of over 150° C. is required for this purpose. The author has found, however, that temperatures approaching the boiling point of water are sufficient provided that suitable apparatus is used.

The work of the author has been carried out in a press, which, from a mechanical point of view, may be seriously criticised, but from a practical point of view is extremely effective and efficient. Experiments on this small press showed that after warming to the boiling point of water, suitably applied pressure rising slowly to half a ton per sq. in. reduced the water-content of Norfolk peat below 50%. In some of the experiments with certain Norfolk peats a figure of 35% was obtained, but generally the figure was 42 to 45%. It will be obvious that the final percentage of water will depend upon many factors, but mainly upon the original vegetation from which the peat was formed and its age.

With peat from the Doncaster district a final moisture content of 55% was found to be the limit,

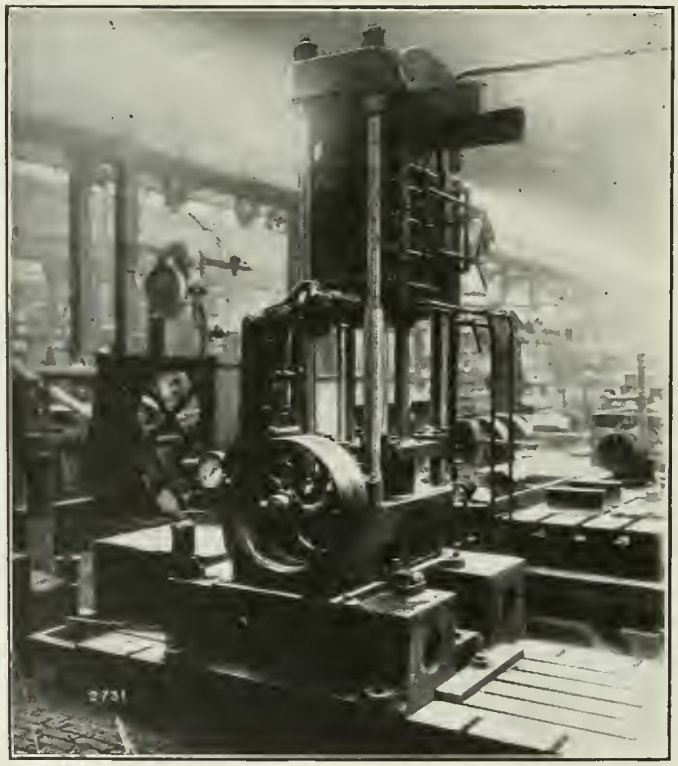


FIG. 2.

and in experiments with peat from other districts in this country it is found that the lower limit for practical purposes varies from about 40 to 55% of moisture.

With regard to the cold pressing of peat, it is desirable to know to what extent cold pressing can be applied, and Fig. 1 is a curve obtained in experiments on Somerset peat, from which it will be seen that at a pressure of nearly 800 lb. per sq. in. and pressing under the very best conditions the percentage of water was only reduced to an average of about 75 %. The press used is shown in Fig. 2 mounted at the works of Armstrong, Whitworth and Co. The stroke of this press was 18 in. and the area of the pistons 120 sq. in., the width of the cake being 5 in.; the head and piston of the press were solid, and the sides of the press were formed of filtering surfaces consisting of tinned steel wire gauze.

It will be noticed from the curve that the particular peat used on that occasion did not part with any water until a pressure of 150 lb. per sq. in. had been reached. On examination of the press after the cold pressing was concluded, it was found that the filtering surface was completely choked, and that a considerable amount of work in cleaning was necessary before the press could be used again.

As already stated, the sides of the chambers were provided with filtering material, and the mechanical construction of this part of the press calls for a great deal of ingenuity in order that the filtering surfaces of large presses can be renewed without much loss of time. The pistons slide on strips of metal which are used to hold the filtering surfaces in position, and at the same time protect the filtering surface from wear from the piston itself. Now it will be noticed that in this method of working the direct pressure of the press does not come upon the filtering surface. During no part of the process of pressing does the peat behave as a fluid, and only to some extent does it behave as a semi-fluid. There is little doubt, however, that near the end of the stroke of the press the lateral pressure is not more than one-third of the vertical pressure, whilst at the beginning of the pressing the lateral pressure near the piston is about four-fifths. Arrangements are made in the press so that steam

Curves showing change in pressure, thickness of cake, flow of water.

Charge 38 lb. 54·4 to 80%.
Cakes 19 lb. 54·4 to 59·0 %.
Steamed for 2 mins.

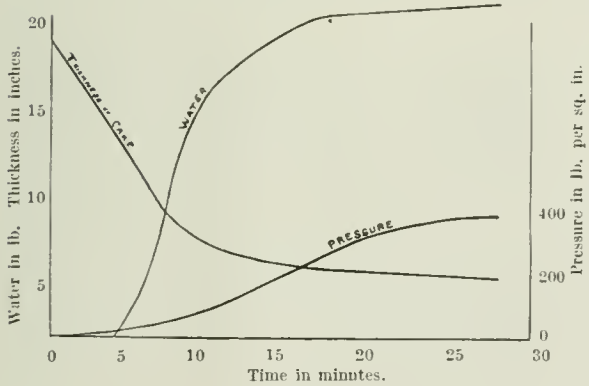


Fig. 3.

may pass through one filtering surface and discharge through the opposite filtering surface and *vice versa*. At the same time when the press is full of peat the admission of steam may be used to blow out any accumulation of deposit at the back of the filtering surfaces. It will be realised, therefore, that the surface of the peat nearest the filtering surface is submitted to the highest temperature conditions, and that normally in a pressing operation the colloidal matter in the centre of the cake is only partially destroyed, while that nearest the filtering surface is wholly destroyed.

The operation of pressing in this little press may be completed in from 20 mins. to half an hour, and in the first two minutes of this operation the steaming may take place. Visible steam is not produced. The curves (Fig. 3) show a typical hot pressing of Somerset peat by which the percentage of water was reduced from 80% to about 57%. On comparing the curves of Fig. 3 with Fig. 1 it will be noticed how the water curve lies much above the pressure curve in the latter case, and it will also be realised that the pressure necessary is now less than 500 lb. per sq. in. Now the reduction of water content from 80 to 60% means that one part of peat associated with four parts of water has become one part of peat associated with one and a half parts of water, that is, by this process of pressing two and a half parts of water associated with the peat have been eliminated. The cost of a press and its wear and tear are not such serious matters with pressures up to 500 lb. per sq. in. as with pressures formerly considered necessary. The reduction of the water content from 55 or 60% to 25 or 30% is not a very serious problem, although at first sight it would appear so. To begin with, the peat cakes are discharged from the press at a temperature of about 95° C., and in cooling down to the ordinary temperature while exposed in a suitable way to the air, the actual water content is reduced nearly to 50%. The further drying to 30% (the usual air-dried peat figure) may be carried out in the course of 4 or 5 hours by passage through a long tunnel dryer the air of which is heated by the waste heat from the power plant.

A simple estimation of the amount of energy required to carry out this pressing operation is desirable. In the first instance, at the factory one part of peat is associated with four parts of water, and it may be taken that to heat this material to 100° C. about 320 calories per kg. of dry peat present would be required. Assuming that the dried peat had a calorific value of 5000 calories per kg., this means that between 6 and 7% of the energy of the peat is required for this process. If, however, air-dried peat with a calorific value of, say, 3500 calories is being burnt in the power plant, this would be equivalent to 9% of the heat energy available from that peat. Assuming that the power plant has an efficiency of 50%, which ought to be a sufficiently conservative value, this would mean that 20% of the energy of the peat would be required for the steaming process.

The cost of the pressing processes in heat energy is readily reckoned, and will be found to be approximately 0·7% of the heat energy contained in the peat. Since, however, hydraulic plant is notoriously inefficient, 7 times this value may be taken as a figure certain to be realised, and it may be assumed that not more than 5% of the energy of the peat will be needed for the pressing operations. These considerations show that one quarter (20%+5%) of the peat produced must be burnt in the power plant to produce the energy necessary for the process, three-quarters of the production being available for the market or other purposes.

The little press illustrated has been worked for six weeks continuously without any difficulty with the filtering surfaces and with practically no variation in its performance. It was designed after several years of experiment as a small portion of a larger commercial press of six chambers to take a charge of 1½ tons per operation. Such a press with accessories would cost about £2000, and would treat

about 60 tons of 80% peat per day of 24 hours, yielding 30 tons of peat cake and about 20 tons of dried peat per day, of which 5 tons would be burned in the power plant, giving a net yield of 15 tons per day.

The commercial efficiency of this process is bound up with that of the method of obtaining peat from the bog.

This subject is an extremely wide one, but it may be assumed that with a project of reasonable size, the modern excavator or a modification of the dredger would give that low cost of winning from the bog that is necessary.

The present wages cost of cutting peat on the peat moors of England by hand-labour amounts to approximately 80d. per ton of product, viz., air-dried peat. Since, however, at the present time it is common to find such peat lying as long as two years before being removed from the drying ground, the total cost of producing air-dried peat must be very high. Peat-cutters are usually skilled workmen aud are paid at a very good rate (approx. 10s. per day). The figure in Somerset is £23 per acre, the acre being only a small portion of the total peat present, the actual amount obtained per acre being about 60 tons of air-dried material.

The application of simple machinery to this process as operations become greater would mean at once a reduction in wages cost of about half that given, and the use of dredgers and excavators would reduce this cost to about one-eighth of the hand-cutting figure. It is obvious, therefore, that the actual cost of production of dried peat will depend very largely upon the outputs determined upon.

Careful estimates made of the cost of production of peat, taking reasonable figures for the winning process at the bog according to the size of the operations involved, reveal the fact that the cost of dried pressed peat by this process would mean, in the case of a one-press plant, 17s. 6d. per ton; with three presses, the figure is immediately reduced to 7s. 3d.; with five presses 6s.; whilst with a ten-press plant the cost of production would be slightly less than 4s. 6d. It would be possible, therefore, to sell such dried peat at the factory at 15s. a ton.

One of the drawbacks of air-dried peat is the fact that its bulk density is so low. This criticism does not apply to peat which has been dried by pressure. In this case the density is usually raised nearly to that of water—about 60 lb. per cubic foot. The density will vary somewhat with different peats on account of the fact that, although the pressure used increases the density considerably, a further contraction takes place in the final drying operation, and this is determined by the residue of colloidal material present in the cake. This increased density is particularly valuable and facilitates the manufacture of household fuel or of peat charcoal from the product. Dried peat possesses a definite "equilibrium moisture" which varies according to its origin. On the peat bogs themselves the "equilibrium moisture" is generally in the neighbourhood of 25%, while in London the figure is approximately 15%. A good fuel should be as free from moisture as possible, and many methods have been proposed for semi-carbonisation to be followed by a briqueting process by which a permanent dry fuel can be obtained.

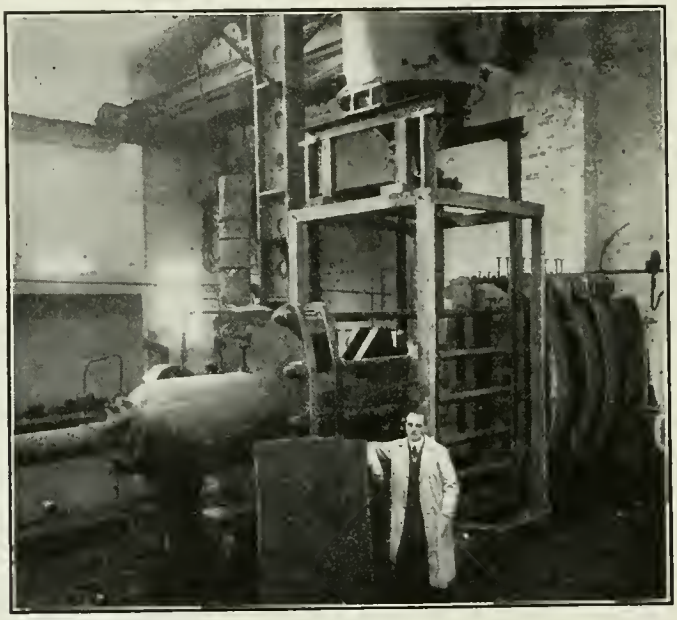


Fig. 4.

That this process of pressing can be carried out on a large scale has been demonstrated by means of a large press which was erected at Stockton two or three years ago. This press was designed to handle slurry peat; the chambers had a width of 4 in., a height of 4 ft. 6 in., and a stroke of 3 ft. 9 in. Fig. 4 is a photograph of the press as it appeared at the works of the Power Gas Corporation, Stockton-on-Tees. The press was intended to extrude the material after pressing so that it should be continuous in operation. It was found, however, that the rate of working the slurry peat was too slow on account of the immense amount of water which had to be driven through the filtering surfaces. The press was therefore worked by the method already described, for which it was not well adapted, that is, semi-wet peat was treated. This operation was remarkably successful, aud peat cakes were produced from Norfolk peat having a moisture content of 45%.

Discussion.

Dr. R. Lessing said that from the nature of peat it was not likely that they would be dealing with water at all but with a solution of salts and also of organic matter, particularly colloidal matter, and it seemed to him that the nature of that solution must have a very great bearing on the possibility of freeing the peat from water.

Mr. P. Parrish said that in connexion with the sale of bog ores and oxides of iron, the practice had been adopted that all expressions should be based on the dry basis in order that a true indication might be arrived at as to the moisture content. Referring to some peat deposits about 40 miles from Melbourne, where there were something like 23 million tons of peat of a most peculiar structure and origin, Mr. Parrish said it existed quite adjacent to the sea and might be described as a vegetable peat which was a kind of guano; when dried and distilled it gave from 18,000 to 19,000 B.Th.U. per ton and a low grade gas of 250 B.Th.U. per cb. ft., and about 100 to 120 lb. of sulphate of ammonia per ton, and yet it was impossible to work this material on an economical basis simply by reason of the peculiar structure and the way in which water appeared to be occluded in certain colloidal matter. Humic acid occurred, and it was a matter to which attention should be directed. Could Professor Hinchley say anything with regard to the de-watering of the top layers of peat which was interesting a good many people in this country?

Mr. Colin Sutton asked if the process described in the paper could be used for dealing with sewage sludge.

Mr. Radcliffe, secretary of the Manchester Section of the Society of Chemical Industry, said he had some experience with Irish peat from the North of Ireland, and the experience there was that the humic acid content of peat differed according to the depth to which the peat was worked. It was also found that the liquid from the peat varied according to the depth. Further, there was also a very considerable difference in the preservative properties of peat according to the depth from which it was taken; Irish peat possessed most extraordinary preservative properties.

Mr. A. J. Broughall asked if the principle of vacuum drying had been applied to this problem, because it seemed to offer great possibilities. He wondered if the question of air bubbles had been considered, because it seemed to him that in peat a great deal of air must be locked up, and the existence of these air bubbles on the edge of the cake might retard the flow of water from inside.

Dr. Reilly, speaking with regard to the disinfecting nature of the material from peat, said that before the war at the Portadown works for the production of producer gas from peat, Professor Morgan obtained a large number of peat tars which contained phenolic bodies which were quite different from what was obtained from coal. Work on these was not continued, and he did not know whether these disinfecting bodies were in the original peat.

The Chairman said he had hoped that some questions would have been asked about the heat pump. As to the actual pressing, he understood that it was very difficult to get equal pressure all over the surface of the cake, particularly a cake of the size dealt with in this case, and he would like to know if any careful investigations have been made to find out the percentages of moisture as they were distributed throughout the cake, because he should imagine that there would be portions relatively dry and other portions containing a considerable amount of moisture. A press had recently been invented by one named Humphrey—he believed a brother of the inventor of the Humphrey pump—which gave equal pressure all over, and it was entirely different from any press he had seen. Some remarkable results had been shown to him with this press, and the great claim for the press was that it gave absolutely equal pressure all over the cake.

Professor Hinchley, in reply, stated that there must be a variation of moisture in the cake, for many reasons. The application of pressure to the material by filtering surfaces would cause a flow of liquid through these filtering surfaces. On account of consolidation through pressure and loss of liquid and variation of pressure in the cake through internal friction, and also the fact that equilibrium is never obtained, the moisture in the middle of the cake may be over 5 per cent. greater than that near the sides. One thing must be avoided, namely, that the rate of flow through the outer layers be not greater than the rate of flow from the middle of the cake, or the formation of an impervious crust will take place and the operation will fail. On this account the rate of increase of pressure needs careful adjustment and control. The question of securing a uniform pressure all over the cake was a matter well worth consideration; on the other hand, it was one of those minor points which did not affect the work very seriously. He worked with a pressure of from 400 to 800 lb. per square inch, and the variation in pressure from the top to the bottom of the cake was in the neighbourhood of 50 lb.

The most economical way of working in the present case was to use a maximum pressure of 500 lb. per square inch. The press mentioned by the chairman worked with toggle joints. The difficulties with such a press were far greater than the difficulties introduced by the ordinary hydraulic press. Hydraulic presses on this work had been in operation for three years and the differences in the results were negligible.

As to peat tars these, he was convinced, would become a big commercial proposition in a few years.

He was not particularly enamoured of vacuum drying, and from the results he had seen he had come to the conclusion that vacuum drying was not quite the wonderful thing that it might appear. As to variation in quality with the depth of the peat, mentioned by Mr. Radcliffe, the last layer of peat was a slurry, and it was better to leave it. A good reason for not taking out the lowest layer was that by doing so the ground was not so readily available again for agricultural purposes. Most of the experiments were done with peat 6 ft. or 8 ft. down. As to sewage sludge, he had not gone into that, but it appeared to be much the same as the lower layers of peat to which he had referred. With regard to the terminology in the paper, he did not apologise for the terms he used. The peat mentioned by Mr. Parrish did not come within the scope of the investigations he had been doing and would not work at all. There had been a great number of centrifuges tried for peat, but invariably they had been failures. The "Gee" centrifuge was a larger machine working successfully, but when they considered the putting through of several hundred tons of peat in a centrifuge they would realise the size of the plant required and the capital involved. He did not think the solution of the peat problem lay with the centrifuge. As to working in two stages, as mentioned by Mr. Parrish, the process described in the paper actually worked in three stages. As to the nature of the filter surface, it was a special metallic gauze. The actual loss of peat in the water given off was negligible, but there was a great deal of work necessary in the investigation of the constitution of peat, and for that reason it was best to commence the work on a small scale before dealing with it on the large commercial scale.

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